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Manufacturing Methods and Technology Measure for Arc-Plasma-Sprayed Phase-Shifter Elements

Final Engineering Report

27 June 1975 to 15 November 1977

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Manufacturing Methods and Technology Measure for Arc-Plasma-Sprayed Phase-Shifter Elements

Final Engineering Report

27 June 1975 to 15 November 1977

Object of Study

The objective of this manufacturing and methods technology measure is to establish the technology and capability to fabricate phase-shifter elements by the arc-plasma spraying techniques.

Contract No. DAAB07-75-C-0043

J. J. Green H. J. Van Hook R. J. Maher D. Massé

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ABSTRACT

The arc-plasma-spray (APS) process has been used to fabricate dielectric-loaded phase shifters of a c-band geometry. The ferrite material is a Li-Ti-Mn ferrite with magnetization (4π M_s) of 1200 G and a dielectric constant (K') of 18.7. The dielectric is Li-Ti-Mn-Al ferrite with 4π M_S = 0 and K = 20. An oven arrangement and sample transfer scheme have been developed which allows a production rate of 5 sprayed boules per A production run of 200 samples was made at this rate. For 50 phase shifters measured at 5.45 GHz the differential phase shift was 393° with a standard deviation of 20°. Insertion loss was <1 dB for 24 of the 50 samples and < 2 dB for 35. The insertion phase of the phase shifters showed a standard deviation of 40°, about double the variation found in conventional c-band phase shifters. These fluctuations in insertion phase are attributable to density variations in the ferrite coating the order of ± 3 percent. The coercive force on plasma-sprayed material was $2 \le H_c \le 3.5$ Oe, somewhat larger than the same material when conventionally fired (H_c = 1 Oe), and attributable to the higher porosity of this material.



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PURPOSE

The phased-array radar antenna is now well established as a means of achieving agile search and multi-target tracking in the current and projected military environment. Each new system with its phased-array antenna has many thousands of radiating elements. Since each element contains a ferrite phase shifter, it is appropriate to develop manufacturing methods and processes that will minimize the cost of each phase shifter.

The purpose of this program is to develop a manufacturing capability for producing a c-band phase-shifter element by arc-plasma spraying of a lithium-titanium ferrite onto a dielectric substrate. In this process, a high temperature diffusion bond between the toroidal envelope and dielectric core permanently mates the ceramic parts, thus eliminating the need for any joining material. The switching wires are threaded through interior slots after final machining and can be replaced or renewed at any time. The primary objective is to produce the phase control element as a finished composition with acceptable microwave properties and a reasonably high yield. To achieve sound composites, one of the properties needing constant monitoring is the match in thermal expansion coefficient between the ferrite coating and the dielectric.

A second important area for control and reproducibility is the thermal environment during spraying. Thermal conditions are influenced mainly by arc current, the arc gas and powder gas velocities, and the substrate-to-gun separation distance. The density and uniformity of the ferrite deposit depend on the reproducibility of these parameters and on the spray dried particle size and size distribution of the ferrite material.

Finally, to achieve a low unit cost, it is necessary to improve yield and reduce machining costs by working with local machine shops to improve overall efficiency.

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GLOSSARY

Annealing - A heating schedule similar to firing but performed on a dense material to relieve strain, improve homogeneity or recrystallize a microcrystalline material.

Arc Plasma Spraying - High-temperature deposition technique in which molten or partially molten material is sprayed onto a heated substrate.

Coercive Force - The horizontal displacement of the magnetization vs applied field curve the hysteresis loop at zero induced field. A measure of the energy required to move magnetic domains through a solid material.

Core Material - The dielectric material which fills the hollow space within the ferrite toroid.

Dielectric - Oxide compounds which exibit polarization in electric fields.

Dilatometer - A device for measuring thermal expansion.

Elastic Modulus - The ratio of stress-to-strain (in pounds/in. 2 or Newtons/in. 2) in isotropic materials which gives an indication of the stiffness or resistance to deformation. Also referred to as Young's modulus. Typically 10 to 50×10^6 psi for oxides.

Ferrite - Oxide compounds of iron and other elements that exhibit a spontaneous magnetic moment due to magnetic spin dipole alignment within the structure.

Hysteresis Loop Properties - The display of magnetization vs applied field for a toroidal or long rod-shaped sample of a ferromagnetic material. The display, generally obtained or low frequencies (\leq 102 Hz) is useful in predictions of the magnetization properties and phase shift behavior at microwave frequencies (\approx 10¹⁰ Hz).

Firing - Any high-temperature process performed on a material, but usually referring to a heating schedule which transforms a powder aggregate into a dense ceramic.

Isostatic Pressure - A powder compaction technique in which a sealed deformable container (e.g., a rubber bag with powder inside) is subject to a uniform compacting pressure from all sides.

Latched State - State of remnant magnetization after application of an applied field sufficient to magnetize in one or two opposite (180°) directions.

Lithium Ferrite - A class of ferrite materials with the general formula $\overline{\text{Li}_{.5} + \text{x/2}_{.7}}$ $\overline{\text{Ti}_{x}}$ Zny2 Fe_{2.5} - 3x/2-yO4 characterized by a saturation magnetization of 0 < 4 π M_S < 3600, a dielectric constant 18 < K < 20, and frequently used in microwave devices.

Magnetic Compensation - A condition obtained in a specific ferrite composition and/or at specific temperatures where the magnetic moment is zero. At this point the opposed magnetic sublattices within the single phase composition exactly compensate.

Magnetometer - A device for measuring magnetic moment.

Microwave - That part of the electromagnetic spectrum between 100 MHz and 100 GHz.

Phase Shifter - A microwave device which serves as the active element in phased-array radar systems where the state of magnetic polarization is used to control the phase length of the electromagnetic energy. Also called phase control element.

Remanent Magnetization $(4\pi \, \mathrm{M_r})$ - The value of induced field remaining in a material with toroidal geometry at zero applied field following the application of an applied field sufficient to uniformly magnetize a material.

Saturated Magnetization $(4\pi \, \mathrm{M_S})$ - The saturation magnetization (c.g.s.) is the magnetic moment gauss/cm³ of a material in an external DC field of sufficient magnitude to align the magnetic moment in the material parallel with it.

Saw Kerf - That portion of a solid removed by the cutting blade. The kerf width is usually about 5 percent wider than the width of the blade.

Scanning Electron Microscopy (SEM) - An instrument using electron excitation and emission to produce images at high magnification with good depth of field.

Spinel Ferrites - A class of iron oxide compositions having face-centered cubic crystal structures similar to the mineral spinel (MgAl₂O₄) and a magnetic moment which depends on composition.

Spray-Dried Powder - A form of powder aggregation where spherical particles of ~ 10 to $100~\mu m$ are produced which are themselves aggregates of much smaller ($< 1~\mu m)$ particles. The advantage of this process is that the aggregates have better flow properties than untreated powder. The process is accomplished in a spray drier, a large funnel-shaped cavity into which a liquid suspension is sprayed and dried.

Stoichiometric - The idealized atomic proportions of elements in a chemical composition, such as the 1:2 in Mg:Al ratio in MgAl₂O₄. Departures from the exact integral proportions may have important effects on properties.

Stress-to-Failure - A statistical or average stress level of a solid where failure by brittle fracture propagation takes place, also called the modulus of rupture. Depends on surface conditions as well as intrinsic strength.

Thermal Expansion Coefficient - A parameter denoting the change in dimension $(\Delta \ell/\ell_0)$ per unit temperature between ambient conditions and some elevated temperature. Since the actual expansion is not perfectly linear, one must specify the thermal interval of interest; i.e., $\alpha \frac{1000}{2000}$ ° = 15 ppm °C⁻¹ denotes expansion between 20°C and 1000°C has our average slope $\Delta \ell/\ell_0 \Delta T$ of +15 × 10⁻⁶ in./in./°C.

Toroid - A ring-shaped or hollow rectangular tube specimen used in magnetic measurements, particularly the hysteresis properties.

X-Ray Analysis - Analysis of crystal structure (X-ray diffraction), elemental composition (X-ray fluorescent analysis) to control processing or elucidate property variations using short wavelength radiation.

1.0 INTRODUCTION

1.1 History of C-Band Phase-Shifter Elements

Ferrite components were used in radars long before phased-array antennas. The idea of obtaining differential phase shift by placing small slabs of ferrite material at the planes of circular polarization in a wave-guide originated in the 1950's. Differential phase-shift circulators made with permanent biasing magnets located outside the waveguide have been used for more than 20 years. However, these devices have never required particularly tight materials property tolerances, nor have they been significant contributors to overall system cost.

In the early 1960's the differential phase-shift circulator geometry was modified to make a latching-type variable phase shifter. The permanent biasing magnets were removed and the flux path was closed inside the wave-guide by using a toroidal cross-section. The inclusion of many thousands of these devices in a phased-array antenna has posed a severe challenge to the ferrite materials properties, and has heightened the impact of the phase shifter on systems cost.

Unfortunately, at C-band and below, a large volume of ferrite material is required for a single phase shifter. In addition to the large volume, this material cost has been aggravated by the need to use the expensive rarearth garnet materials to achieve low insertion loss and acceptable temperature performance in devices operating below 6 GHz. In the mid-1960's Temme, Ince, and Stern (1967) pointed out that a high-dielectric constant nonmagnetic material, inserted into the magnetic toroid, would significantly reduce the required dimensions of both the ferrite and waveguide. This reduction of the ferrite volume made it possible to consider the expensive garnet materials for use in low-frequency (< 6 GHz) phased arrays.

Present c-band phased-array antennas tend to use a garnet toroid with a dielectric insert for the phase-shifter element (Fig. 1). The rectangular toroid is 5.145 in. long, 0.250 in. high, 0.220 in. wide, with 0.050 in. walls. The dielectric insert is barium tetratitanate (also called K-38 because its dielectric constant is 38). The toroid is formed around a steel pin, then fired to a dense ceramic. The dielectric is formed and fired as 1.5 kg bars. Each bar yields about 40 inserts machined to the final dimensions. Since the toroid is an as-fired piece with a center hole which cannot be guaranteed to be absolutely straight or uniform in cross-section, the insert is undersized (0.109 in. by 0.139 in. cross-section) and is coated with a resin material just before it is inserted into the (0.120 in. by 0.150 in.) toroid opening. Grooves are machined on each side of the insert to allow for the three copper wires used to switch magnetic polarization. In assembly, the wires are fitted into the slots, the mating surfaces and wire slots are coated with resin, the two parts are forced together, and the composite of toroid insert wire and resin is put through a complex thermal cycle to cure the resin.

1.2 Difficulties with the Current Approach

While many tens of thousands of phase shifters have been fabricated for various phased array antennas, the manufacturing process could be improved both to reduce cost and to enhance antenna performance. The present process has several disadvantages. First, it is expensive to assemble the tight-fitting component parts. Second, the resin material does not have completely reproducible curing characteristics, and requires that the curing cycle be varied from run to run. Third, the switching wires are bonded permanently by the resin. If a wire should break during manufacture or in later use, the entire unit must be scrapped.

These three problems add to device cost. However, there is also evidence that the resin can seriously affect phase-shifter performance, causing a variation in insertion phase from one unit to the next. This variation could could cause the radar beam to broaden and the sidelobes

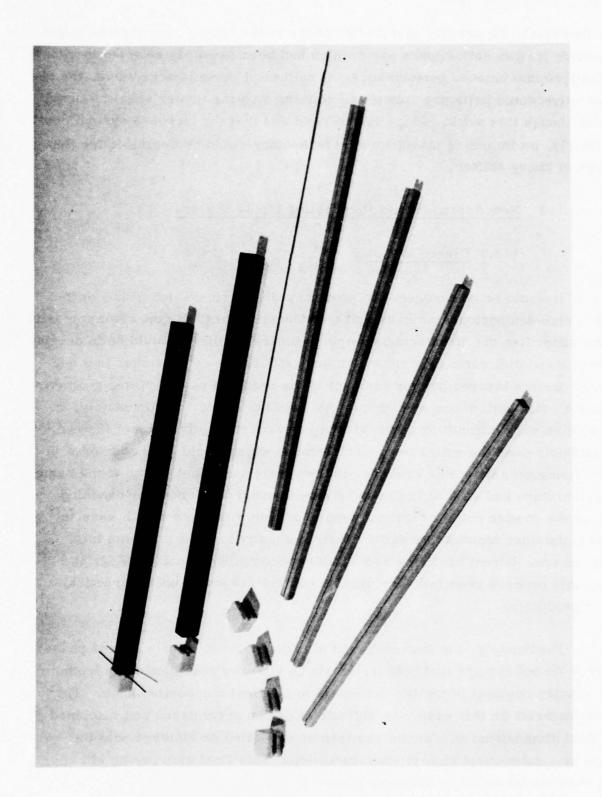


Figure 1 K-38 Dielectric Rods, K-16 Dielectric Spacers, and Ferrite Toroid Manufactured at Raytheon

to increase. To prevent degradation of the radar beam, each antenna might require its own set of spare parts which had been carefully selected to maintain optimum antenna performance. If neither of these is acceptable, the alternative would be to use only phase shifters meeting tighter specifications, even though this would reduce device yield and thereby increase system cost. Clearly, an improved manufacturing technology would be desirable for this type of phase shifter.

1.3 New Approaches to Fabricating Phase Shifters

1.3.1 Direct co-firing

It would be advantageous to produce a dielectric-loaded phase shifter by a high-temperature process that eliminates the need for the resin material and simplifies the manufacturing steps. A direct solution would be to develop ferrite and dielectric powders which are sufficiently well matched that one could form a layered powder compact in the required phase-shifter geometry, then co-fire both in one step to the final dense ceramic. Unfortunately, it would be very difficult to match all the relevant characteristics of the two materials over the entire temperature range which would be encountered in the firing process. For example, the two materials must sinter at the same temperature and at exactly the same rate to avoid distortions or cracking. Also the powder compact (green) density of both materials would have to be maintained identical as would the fired density to yield the same total shrinkage. Direct co-firing was actually accomplished in 1974 (Fig. 2), but this process required very precise control and would not be practical for production.

Fortunately, the materials and dimensions needed for a c-band phase shifter do not require that both materials be sintered simultaneously from the powder compact of the two materials to the final composite shape. The core material (in this case, the dielectric) can be presintered and machined to final dimensions; the ferrite can then be deposited or sintered onto the core by a subsequent high-temperature step. This final step can be either arc-plasma spraying or firing-in-place.

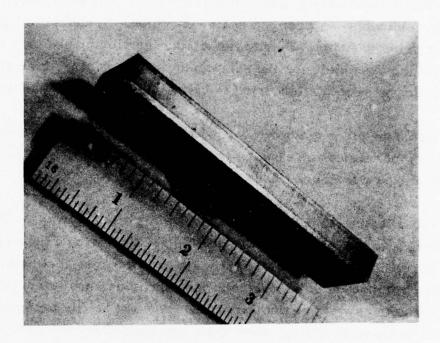


Figure 2 Photograph of Layered Composite with Outer Ferrite Layers (1250) and Dielectric Core.

1.3.2 Co-manufacturing by firing-in-place

At the start of the arc-plasma-spraying outlined in this report, the firing-in-place technique was under development at Raytheon for only a few months. As it has since evolved, the process is very similar to arc-plasma spraying in that both methods make use of similar dielectrics and are fired on at similar temperatures. In firing-in-place a conventionally processed ferrite powder is isostatically pressed into a toroid, just as is done for current phase shifter elements. A prefired and premachined dielectric is inserted immediately prior to firing. During firing, the ferrite shrinks onto the dielectric, leaving open slots for the subsequent insertion of switching wire. Some machining is needed after firing to bring the element to final dimensions.

The firing-in-place method has not yet reached the stage where the reproducibility of tens of units can be tested, but results on individual samples have shown some promise.

A critical factor in developing the firing-in-place, as well as the cofiring method, was the fabrication of spinel-type dielectrics with a range in dielectric constant(k') and thermal expansion coefficient $(\bar{\alpha})$ for the matching of $\bar{\alpha}$ and the optimization of k' relative to the ferrite composition. While the spinel dielectrics were developed before the start of this program for experiments in direct co-firing, they were also needed for the APS program outlined in this report.

1.3.3 Arc-plasma spraying

The arc-plasma-spray (APS) process was first applied to the production of microwave phase shifters by R. Babbitt². It was already a well-established process for refinishing critical metal parts with wear-resistant, temperature-insensitive coatings. This process can be used to spray low-melting-point (aluminum) or refractory (tungsten) metals or metal oxides because of the wide latitude in latent heat transfer from the very hot plasma to the particulate feed material.

Babbitt et al.^{2,3} were the first to apply the APS technique to electronic materials. In the process developed by Babbitt, ferrite powder is partially melted by an intense plasma heat source and deposited at high temperatures (1300°C) in dense, microcrystalline form onto a dielectric substrate whose thermal expansion matches that of the ferrite. The phase-shifter boules are sprayed in a single axial pass with rapid (100 rpm) rotation in a 750°C oven. They are later heated to 1015°C to optimize the dielectric and magnetic properties. A machining step then removes the excess ferrite, and a final anneal (to remove the machining stress) completes the manufacturing process.

Before this program began the ferrite powder used in the initial experiments at ECOM and Raytheon was a lithium-titanium-manganese ferrite developed for conventional ferrite processing. This powder has been generally satisfactory for APS, although certain additives to the powder (i.e., binder content, $\operatorname{Bi}_2\operatorname{O}_3$ additive, etc) may not be optimal for the latter. The development of these compositions both at Raytheon and elsewhere has been directed toward a replacement for the more expensive garnet materials.

In developing the ferrite material, our goal was to produce a material with a high dielectric constant, magnetic properties which are stable with temperature and insensitive to stress, as well as lower materials cost. The Li-Ti ferrite does meet all of these requirements in conventionally fired form. Babbitt was able to show that these same compositions, when plasma sprayed and annealed under appropriate conditions, would also yield microwave properties that compare favorably with existing garnet materials. Having succeeded in reproducing Babbitt's results in our laboratories, we concluded that there was no intrinsic materials limitation to replacing the current garnet with a plasma-sprayed Li-Ti ferrite. Of course, properties required some improvement and yield and production rates were unknown, but in general the prospects were favorable.

Our experience with the plasma spray equipment was limited to about six months' work using a furnace geometry that was clearly inappropriate

for production. However, one could see at this point that the dielectric-loaded phase shifter geometry was well suited to the APS process. The cross-sectional area of the dielectric (0.120 by 0.150 in.) is small enough to be heated rapidly without thermal shock and large enough to support the extra weight of the plasma-sprayed ferrite coating. Dielectric loading had also reduced the necessary toroid wall thickness (0.050 in. required) to the extent that an adequate ferrite coating along the five-inch long dielectric could probably be deposited in 10 to 15 minutes of spraying time. Assuming that the transfer time between sprayings could be shortened to half this spraying time, the desired production rate of 40 per day could be achieved with one station and one operator.

This report will describe in detail the program to develop manufacturing methods for the production of such phase shifters.

2.0 PROCESS, EQUIPMENT, AND TOOLING OF ARC-PLASMA-SPRAYED PHASE SHIFTERS

2.1 Ferrite Powder Development

The characterization of ferrite powders used in the APS process has become one of the most important controls in the MMT program. With the benefit of hindsight, it is clear that the choice of a composition that would give the required magnetic properties was sound, but the ferrite particle size measurements and process controls were not comprehensive enough to completely characterize subtle changes in the powder that led to important differences in APS behavior and to differences in coating density. Nevertheless, we did make serious efforts to standardize processing and to characterize the ferrite powder as completely as possible.

2.1.1 Magnetic properties

The choice of ferrite composition is an essential first step to meeting the phase-shifter performance characteristics set by the garnet material presently used in c-band phase shifters. The saturation magnetization (4 π $\rm M_{_{\rm S}})$ is a primary factor since this affects the phase-shifter parameter of phase shift and magnetic loss. Room temperature data on $4\pi\,\rm M_{_{\rm S}}$ versus Mn and Li + Ti content are shown in Fig. 3. The inset above the main figure shows the effect of Mn addition to pure Li-ferrite (x = 0). The main figure shows the influence of Mn substitution combined with Li-Ti. Mn addition raises $4\pi\,\rm M_{_{\rm S}}$ in all compositions. This indicates either a preference of Mn $^{+3}$ for the A sites or a Mn substitution on B sites which displaces some Li from B to A, thus increasing $4\pi\,\rm M_{_{\rm S}}$.

Zinc substitution in Li-ferrite and Li-Ti-ferrite raises $4\pi \, \mathrm{M}_{_{\mathbf{S}}}$ for $0 \leq \mathrm{Zn} \leq 0.35$. At higher concentrations $4\pi \, \mathrm{M}_{_{\mathbf{S}}}$ decreases due to a weakening of intersublattice exchange, aided by the very rapid decrease in $\mathrm{T}_{_{\mathbf{C}}}$ with Zn content. The Li and Ti substitutions also lower $\mathrm{T}_{_{\mathbf{C}}}$, although not as rapidly as Zn addition. Table 1 shows data for Li-Ti and Li-Ti-Zn ferrites on the temperature coefficient of the magnetization $\Delta \mathrm{M}_{_{\mathbf{S}}}/\mathrm{M}_{_{\mathbf{S}}}\Delta \mathrm{T}$ between

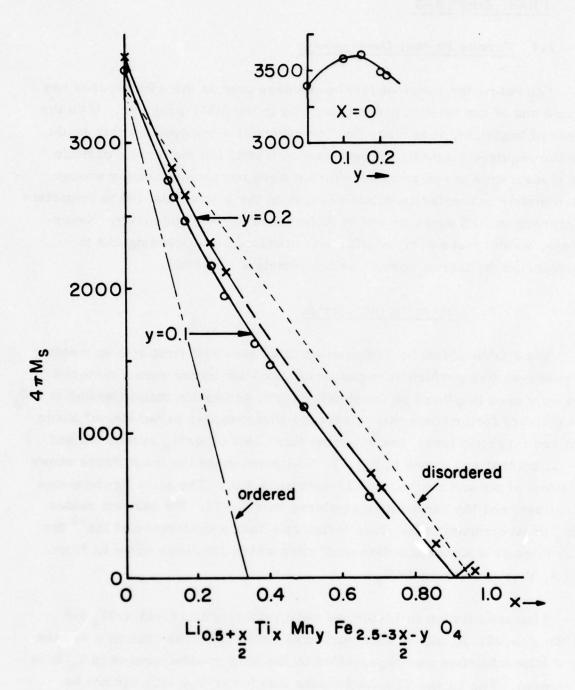


Figure 3 Magnetization versus Composition at 20°C for $\text{Li}_{0.5} + \frac{x}{2} \text{Mn}_{y}^{\text{Ti}} x^{\text{Fe}} 2.5 - \frac{3x}{2} - y^{\text{O}} 4$.

 $20\,^{\circ}$ C and $120\,^{\circ}$ C relative to the $20\,^{\circ}$ C value and estimates of $T_{_{\hbox{\scriptsize C}}}$. In this example these materials contain a constant Mn level of 0.10 per formula unit to control dielectric loss.

The magnetization curves are shown for the first three compositions in Table 1 in Fig. 4. The slope of the curves near 20°C changes only slightly with composition. Thus the percentage increase in coefficient depends on the decrease in $4\pi\,\mathrm{M}_{_{\rm S}}$ due to the reduction in $\mathrm{T}_{_{\rm C}}$. In the last example (a zinc-containing 1250-gauss material), combined substitutions of Zn and Li-Ti have reduced $\mathrm{T}_{_{\rm C}}$ and raised the temperature coefficient substantially.

TABLE 1

CURIE TEMPERATURE OF SEVERAL Li-Ti FERRITES

Saturation Magnetization	Zn Content	Mn Content	Ti Content	T _c (°C est.)	Temp. Coeff.
3600 G	0	0.10	0	625	0.09
2250 G	0	0.10	0.26	525	0.13
1250 G	0	0.10	0.50	390	0.18
1250 G	0.10	0.10	0.54	310	0.27

The results shown in Table 1 indicate that Zn substitution should be kept to a minimum because of its very strong effect on $\mathbf{T}_{\mathbf{C}}$ and thereby on the temperature coefficient of $\mathbf{M}_{\mathbf{S}}$. The reason for the rapid loss of temperature stability is that Zn substitution requires additional Li and Ti substitution to bring $4\pi\,\mathbf{M}_{\mathbf{S}}$ back to a given value. The effect of the two substitutions is additive in terms of the depressive effect on $\mathbf{T}_{\mathbf{C}}$. For these reasons we decided not to introduce Zn.

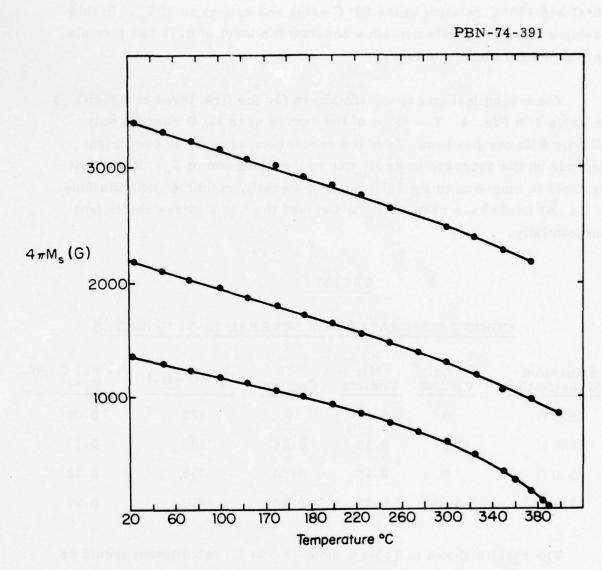


Figure 4 Magnetization versus Temperature for Several Li-Ti Ferrites.

2.1.2 Remanent magnetization

The remanent magnetization (B_r or $4\pi M_r$) of the 1250-gauss Li-Tiferrite is the order of 850 to 900 gauss in dense material. The theories (Grieffer 5) relating B, to ferrite composition stress the importance of the dominance of the magneto-crystalline anisotropy (K1) over stress-induced anisotropy ($\sigma \cdot \lambda_i$) in achieving the highest B_r possible. The Wijn (1954) model theorizes that a maximum B, occurs when domains within the individual grains relax to the nearest [111] easy axis direction for $K_1 \le 0$, a situation possible when K_1 dominates. To minimize the demagnetization energy, Goodenough 6 has proposed the creation of small reversal domains at grain boundaries and near second phases or pores, especially when a local stress is present. The magnitude of B, depends on the number and size of these reversal domains because their collective volume determines how far B_r will be reduced from the theoretical value of B_r = 0.87 M_s for cubic anisotropy. For lithium ferrite the dominance of crystalline over straininduced anisotropy permits relatively large ratios of B_r/M_s, typically 0.70 to 0.75.

The theories relating $B_{\mathbf{r}}$ to ceramic microstructure stress the avoidance of secondary phases and porosity to obtain a maximum $B_{\mathbf{r}}$. Second phase and porosity reduce $B_{\mathbf{r}}$ in two ways: first, by reducing the volume of magnetic material, and second, by providing discontinuities and regions of local strain which favor the creation of the reverse domains.

Since the latching phase shifter calls for $H_c \le 1$ Oe, it is essential to maintain hysteresis loop squareness and thereby high B_r . The first priority should be the development of process controls which will yield a dense, uniform-grain-size ferrite after plasma spraying and annealing.

2.1.3 Coercive force

Coercivity is probably the most microstructure-sensitive of the magnetic properties of importance to phase-shifter performance. It is

influenced both by porosity second-phase content and by polycrystalline grain size. If porosity and second-phase content are kept below 1 percent to satisfy the requirement for high B_r, then coercive force is determined primarily by grain size and anisotropy (i.e., composition).

In addition to the grain size dependence of H_c , many workers ⁵ have reported that Zn rapidly lowers H_c in Li-ferrite compositions. A semiempirical relationship between H_c and material properties reported by Grieffer correlates with these observations:

$$H_c = \frac{\sigma_w}{M_s L}$$
,

where M $_{\rm S}$ is the magnetic moment, L is an average grain size, and σ $_{\rm W}$, the wall energy, is given by

$$\sigma_{w} = 4 \left[A \left(K_{1} + \lambda_{i} \sigma \right) \right]^{1/2}$$

In the second equation, A is the exchange parameter (roughly proportional to T_c), K_1 is the anisotropy constant, λ_i is the isotropic magnetostriction, and σ is the internal stress at the domain wall.

There are two ways in which substitution of zinc reduces H_c . First, Zn enhances grain growth in annealing, which produces a larger polycrystalline grain size. Second, the reduction in T_c reduces both the exchange parameter A and the magnetocrystalline anisotropy constant K_1 , again decreasing H_c . Unfortunately, zinc substitution also leads to a large temperature sensitivity of M_s and B_r and to a rounding of the hysteresis-loop shoulder because squareness depends on a large K_1 . The deterioration in hysteresis loop squareness is particularly bad for the low H_c latching-type phase shifters because it makes it more difficult to reproduce B_r .

Other substitutions such as Co and Mn can also change wall energy through alteration in anisotropy (K₁) and magnetostrictive (λ_i) parameters. One might expect Mn to affect H_c through the magnetostrictive term ($\lambda_i \sigma$)

in the wall energy equation. Our previous studies of Li-ferrite compositions with identical microstructures and different Mn concentrations $(0.01 \le y \le 0.01)$ have shown, however, that Mn content has no appreciable effect on hysteresis loop squareness or coercive force. Therefore, the Mn concentration can be determined entirely by other considerations such as dielectric loss. This behavior contrasts with that of cobalt and zinc, where substitutions change several properties, and a tradeoff must be made.

2.1.4 Particle size

The experimental results of APS runs made with R. Babbitt at ECOM at the beginning of the program (to be described in Section 2.4.1) gave strong indication that spray-dried Li-Ti ferrite powders from Raytheon Special Microwave Device Operations were equivalent to other commercial sources. We chose to use the SMDO ferrite powder, since it gave us direct access to processing history, as well as the opportunity to ask for processing changes if necessary. The first delivery of material, a 34 Kg batch, was made in September 1975. To produce a free-flowing aggregate for the APS gun, the ferrite powder was spray-dried after the final milling step. This treatment is the same as that used to prepare powder for automatic die pressing, where a free-flowing material is essential. In spray-dried form, the particles are aggregates of the much finer (< $1 \mu m$) ferrite powder. A scanning electron micrograph of a spray-dried particle ~30 µm in diameter is shown in Fig. 5. The larger particles are generally hollow, as evidenced in this case by the hole at right center in the spherical agglomerate. The small particles are held together by a small amount (>2 percent by weight) of organic binder.

Particle size control is very important for uniformity in APS melting, since residence times in the plasma flame are the order of microseconds. We requested that the supplier keep the larger spray-dried powder fraction taken from the main chamber separate from the fines fraction which is collected from the effluent in a cyclone separator. Figure 6 shows the results of this first crude separation of the powder into a coarser "chambers

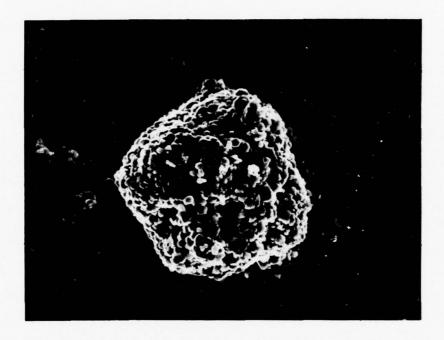
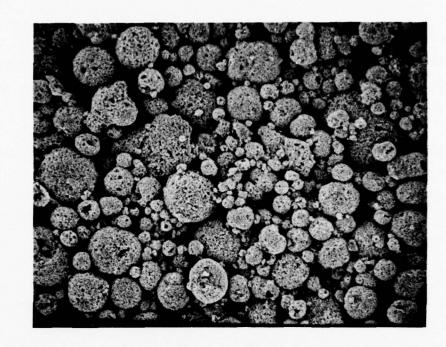


Figure 5 SEM Photograph of Spray-Dried Ferrite Powder at $2000\,\times$.



(a)

(b)

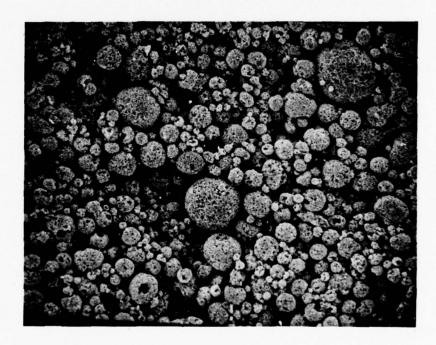


Figure 6 SEM Photographs of Spray-Dried Ferrite Powder (LMTF 53(G2)) at 400×.

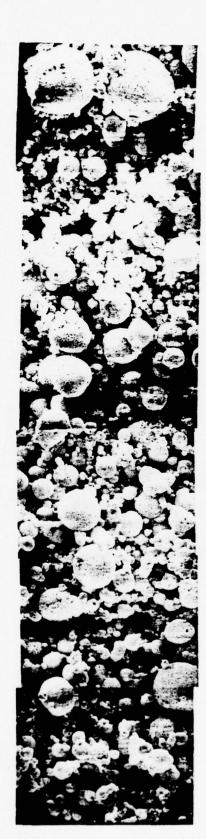
(a) "Chambers fraction"; (b) "Fines fraction".

fraction" (Fig. 6a) and a "fines fraction" (Fig. 6b). The SEM operator was asked to take the photographs at random locations in the two powders. As can be seen, there is a considerable size range within each lot but, nonetheless, some size fractionation between "chambers" and "fines".

Sampling techniques for particle size analysis in powders cover a wide range in sophistication of instrumentation and size of the sample being analyzed. Unfortunately, there seems to be an inverse relation between these two factors. Methods such as the Coulter counter are very convenient and quantitative, but sample only a very small amount of material. Other methods, such as sedimentation and air permeation, sample a larger and possibly more representative portion, but measurements are more indirect and one must rely on assumptions about particle geometry and degree of dispersion that are not always justified. Screening can be used, but this method is rather impractical since spray particles are very fragile and easily broken, unless great care is exercised. Size analysis by counting individual particles is the most reliable method. It can, however, be very tedious to accumulate enough counts for a reliable sampling. Fortunately, the use of a semiautomatic counting device, coupled with the easily resolved size and shape of spray-dried powders, can speed the process considerably.

We have used a Zeiss particle size counting device (described in Appendix I) to generate histograms of the spray-dried particle size of the different ferrite batches. The ferrite powder discussed in conjunction with the particle size counter in Appendix I and shown in Fig. 6 is characterized as LMTF53(G2). This powder in general had poorer flow characteristics than later materials, as for example, the LMTF50(G3) powders shown in Fig. 7 and in the histogram Fig. 8. Note that when all the particles in a photograph are counted, the average size is much smaller than one might estimate visually.

Some of the ferrite powders used in the later APS runs in this program had very good flow characteristics and produced sound phase shifters.



Fines



Chambers

Figure 7 Photographs of Spray-Dried LMTF50(G3) Powder (400 \times).

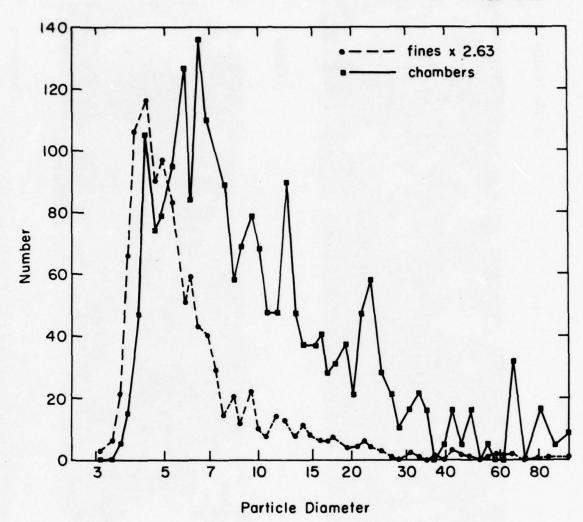


Figure 8 Histogram of Particle Size from Fig. 7. Vertical Scale on Chambers × 2.63.

Two examples were the LMTF475(G5) and LMTF475(G7) powders, both of which had been formulated with less Ti (x = .475) to increase $4\pi M_s$ and compensate for the lower density of APS ferrite (~92 percent of d_x) as compared with conventional firings (~99 percent of d_x) of the same powder.

Figure 9 shows one SEM photograph of spray-dried G5 powder from the chambers fraction and one from the fines fraction collected in the cyclone separator at the exit end of dryer. Figure 10 shows similar SEM photographs of the G7 ferrite powder. The photographs are a collection of six sequential individual photos of a representative region of powder samples taken originally at $400\times$ magnification. Size reduction for publication in this report has reduced the magnification to $175\times$.

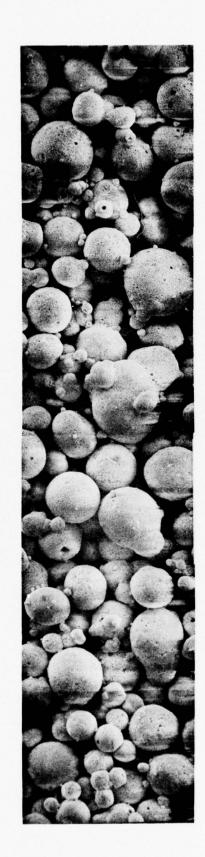
The spray-dried particles in a photograph were counted at the original magnification. To determine the number of counts needed to generate a histogram representative of the sample, we divided the photograph in half and generated separate histograms of the two parts, approximately 800 counts in each. If a doubling of the number of counts does not change the histogram shape, the smaller number is adequate. What is an adequate count is, of course, a subjective evaluation, and we have had to adopt arbitrary criteria to set limits. We have decided that a change in mean particle size of > 20 percent, or radical differences in the shapes of the two distribution curves, would indicate insufficient data for a histogram representative of the powder.

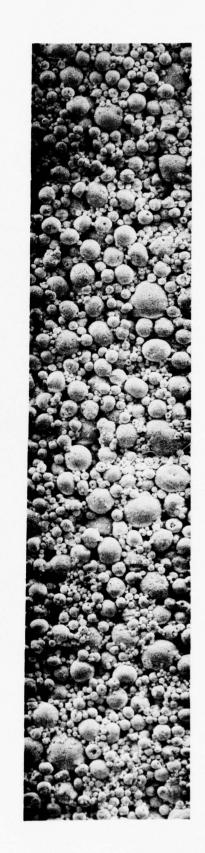
Figure 11 shows two curves for the G7 fines powder fraction - one curve indicating the count in the lower half of Fig. 10; the other, the top half of Fig. 10. Every resolvable particle was counted, totally 1672 different particles of different diameters. The two curves differ in mean value by approximately 15 percent and the particles have similar sizes, indicating that this count is adequate by our standards.



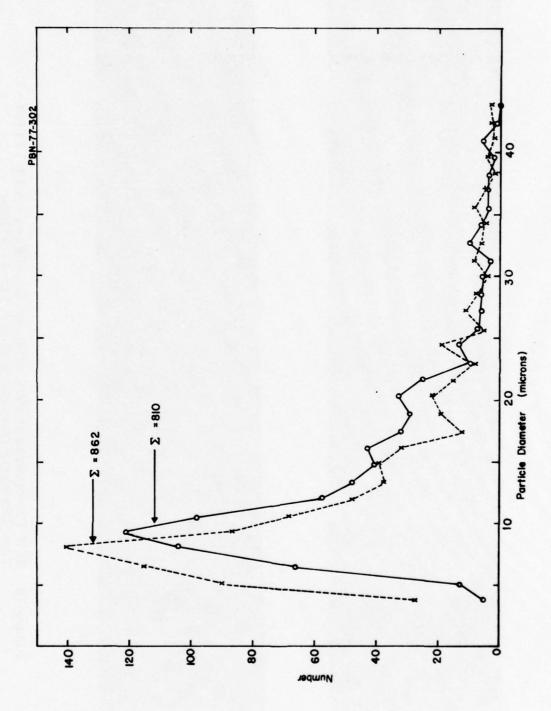


SFM Photographs at $400 \times$ of Spray-Dried Ferrites LMTF475(G-5). Top: Chambers fraction; Bottom: Fines fraction. Figure 9





SEM Photographs at $400 \times$ of Spray-Dried Ferrites LMTF475(G-7). Top: Chambers fraction; Bottom: Fines fraction. Figure 10



Histogram of G7 Fines Powder Fraction Counted on the Lower and Upper Halves of the Photo in Fig. 10. Figure 11

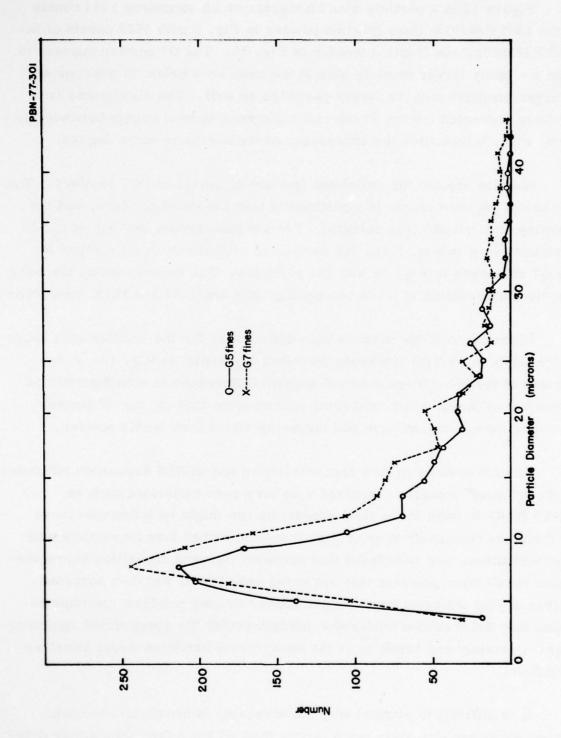
Figure 12 is a particle size histogram which compares 1454 counts of the LMTF475(G5) fines fraction powder in Fig. 9 with 1672 counts of the LMTF475(G7) fines fraction powder in Fig. 10. The G7 powder appears to have a slightly larger particle size at the peak area below 10 microns and a larger proportion of the larger particles as well. The histograms have not been corrected for the 13 percent difference in total counts between powders, which would alter the appearance of the curves to some degree.

We also studied the chambers fraction of the G5 and G7 powders. The number of particle counts is significantly less for these powders, and the counting statististics less reliable. The six photographs making up the G5 chambers view in Fig. 9 had 325 particles, whereas the total number for the G7 chambers in Fig. 10 was 261 particles. The corresponding numbers for the fines fraction in these two photographs are 1454 and 1672, respectively.

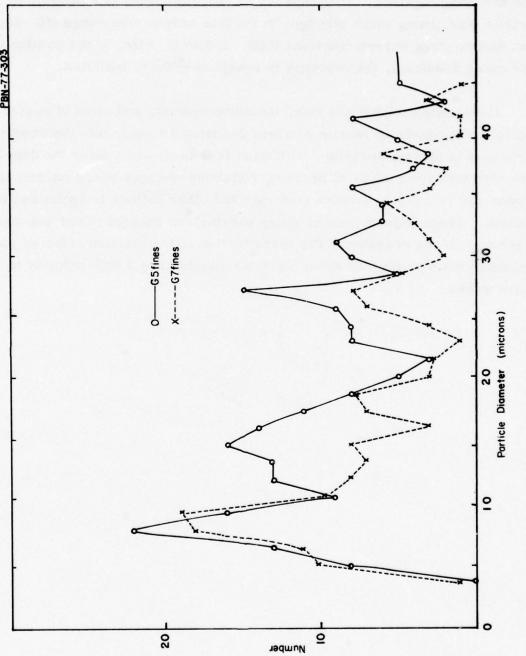
Histograms of the particle size distribution for the smaller size range of the G5(0) and G7(x) chambers fractions are shown in Fig. 13. A comparison of the two curves does not suggest differences in size distribution that seem apparent when comparing photographs; that is, the G7 powder seems to have more uniform and larger particles than the G5 powder.

The differences in flow characteristics and in APS deposition efficiency of these "good" powders compared with very poor materials such as LMTF475(G-8) used in the final production run might be influenced more by moisture content or state of agglomeration rather than by particle size or distribution. We have found that optimum flow and deposition characteristics result from powders that are dried near 100°C and then screened. Higher drying temperatures produce poorer flowing powders, perhaps because they drive off the binder that holds together the spray-dried agglomerates. Certainly any break up in the spray-dried particles would interfere with flow.

It is difficult to pinpoint why the screening is beneficial, because screen sizes are generally much larger than all but a few large spray-dried



Particle-Size Histogram Graphing the LMTF 475(G5) Fines Fraction Powder from Fig. 9 and the LMTF 475(G7) Fines Fraction Powder from Fig. 10. Figure 12



particles. We speculate that the major effect of the screen is not to separate the small particles from the large, but to break up groups of smaller particles that hang together. The tendency to reagglomerate is a function of particle size, being much stronger in the fine screen size range (50 -80 μ m) than with coarser screen fractions (100 - 200 μ m). Also, if the powder is kept under dessicant, the tendency to reagglomerate is inhibited.

Differences in particle size, moisture content, and state of agglomeration in the spray-dried ferrite powders probably all enter into the observed variations in plasma spraying. Different ferrite powders alter the deposit rate anywhere from 20 to 50 percent, requiring changes in arc current and powder gas velocity — powder feed rate and other factors to optimize deposition. These adjustments in spray parameters require talent and ingenuity on the part of the operator. The investigation of the detailed effect of particle characteristics on plasma spray behavior should have a high priority in future work.

2.2 Development of Dielectric Material

The dielectric compositions developed for arc-plasma spraying (APS) are spinel solid solutions with the same crystal structure as the magnetic ferrite. Substitutions of Li and Ti in the spinel can reduce $4\pi\,\mathrm{M}_{_{\rm S}}$ to zero and increase the dielectric constant slightly. While a major concern with these materials is that we maintain magnetic compensation, i.e., that $4\pi\,\mathrm{M}_{_{\rm S}}\approx 0$, the prime need for successful APS production is that the thermal expansion of the dielectric match the ferrite exactly. The LMTF 190 material, with a nominal composition of Li_97Mn_1Ti_95Fe_98O_4, has proved to be a good thermal-expansion match to the 1200 G ferrite. However, this material has a non-zero magnetization: room-temperature values of $4\pi\,\mathrm{M}_{_{\rm S}}=90$ have been obtained. To reduce this residual moment to zero, a series of dielectrics was developed with higher Ti content, to reduce $4\pi\,\mathrm{M}_{_{\rm S}}$, coupled with Al_2O_3 substitutions to bring the thermal expansion coefficient into line with the plasma-sprayed ferrite.

2.2.1 Thermal expansion data

Matching the expansion coefficient of the dielectric is probably the key to successful arc-plasma spraying of ferrite phase-shifter elements. Since the spinel ferrites have both large elastic modulus and small stress-to-failure, mismatch-induced strains must be kept very small over the entire temperature range to avoid fracture.

The expansion coefficient was measured on all of the compositions listed in Table 2. Cylindrical samples between 1.3 in. and 2 in. long were placed in the quartz dilatometer. Programmed heating and cooling rates of 2° C / min were used and the atmosphere was stagnant air. The data are printed out on a computer-controlled x-y plotter as expansion coefficient (α) versus measurement temperature minus ambient (T-A).

Figures 14 and 15 show thermal expansion $\Delta \ell/\ell_0$ (cross symbols) and α (x symbols) versus (T-A) for two samples which had the same dielectric composition but different amounts of Bi₂O₃. (Sample 200 (1) in Fig. 14 had 0.5 wt. percent Bi₂O₃, while Sample 200 (2) in Fig. 15 had 0.1 wt. percent Bi₂O₃). Because of the difference in bismuth additives, the two samples required different firing temperatures, and we wanted to determine whether the additive or the change in firing temperature would affect the expansion coefficient. A point-by-point comparison in $\bar{\alpha}$ reveals differences as large as 0.5 ppm for these two compositions. The differences, however, probably reflect inaccuracy in the measurement rather than intrinsic differences in expansion behavior because identical compositions to be described later show differences in $\bar{\alpha}$.

A series of dielectric compositions were also examined, in which increasing amount of Al were substituted for Fe in an attempt to determine the effect on expansion coefficient. Figure 16 shows $\bar{\alpha}$ vs. (T-A) for a dielectric with 0.07 atom substitution of Al for Fe. In this material $\bar{\alpha}$, at T-A = 100°C, is slightly higher than in the unsubstituted material (Figs. 14 and 15). Figures 17 and 18 represent samples from two different batches which have exactly the same composition, both in terms of major components and ${\rm Bi}_2{\rm O}_3$ content. (The Al substitution, w, is 0.15 in these samples). Differences in data points are again the order of 0.5 ppm, which could be caused either by instrumental error or by differences in sample density.

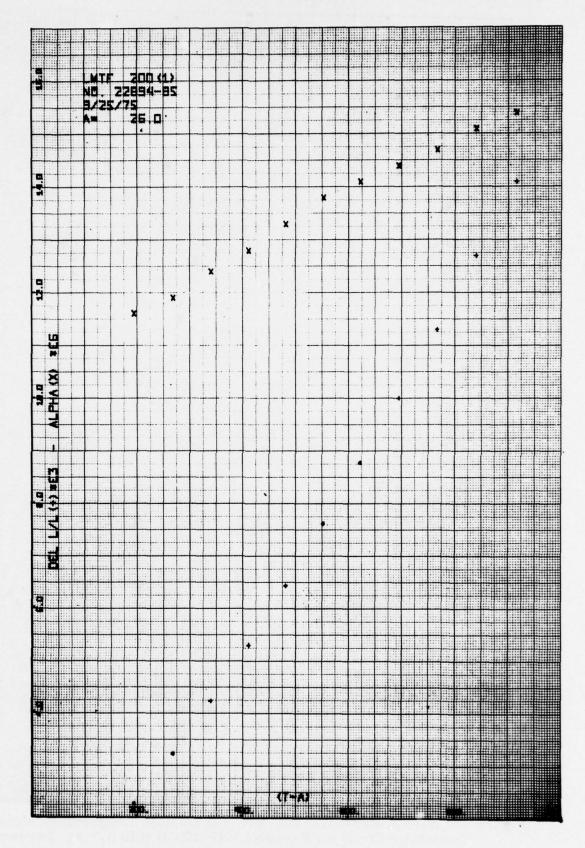


Figure 14 Thermal Expansion (a) vs. Measurement Temperature Minus Ambient (T-A) for Sample LMTF 200(1) with 0.5 wt. Percent Bi₂O₃.

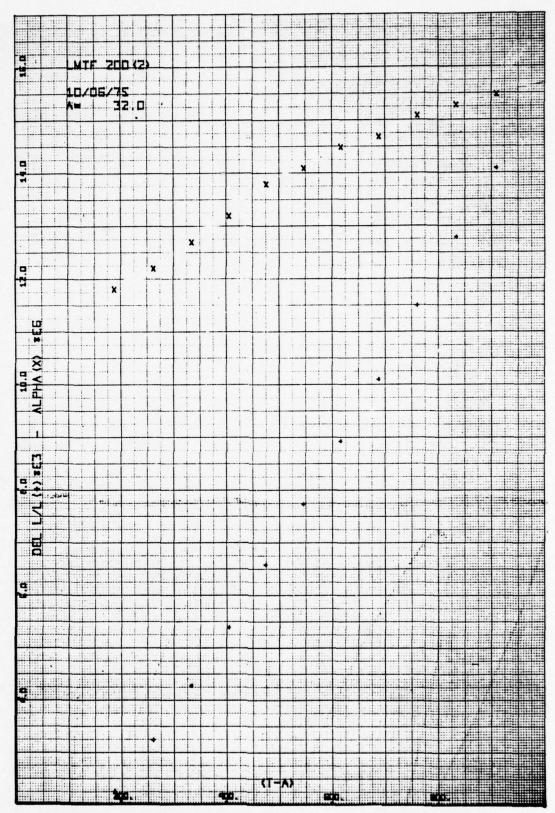


Figure 15 Thermal Expansion (α) vs. Measurement Temperature Minus Ambient (T-A) for Sample LMTF 200(2) with 0.1 wt. Percent Bi₂O₃.

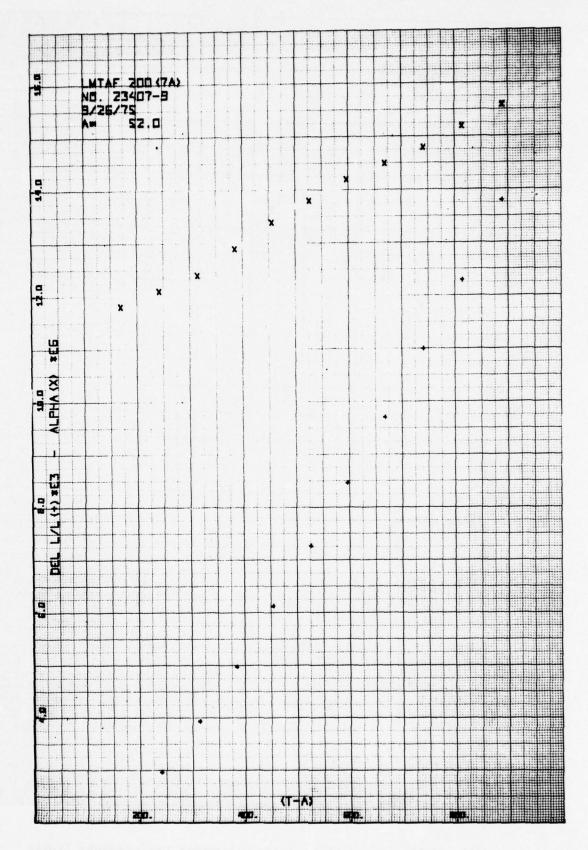


Figure 16 Thermal Expansion (α) vs. Measurement Temperature Minus Ambient (T-A) for Sample LMTF200(7A) with 0.07 Atom Substitution of Al for Fe.

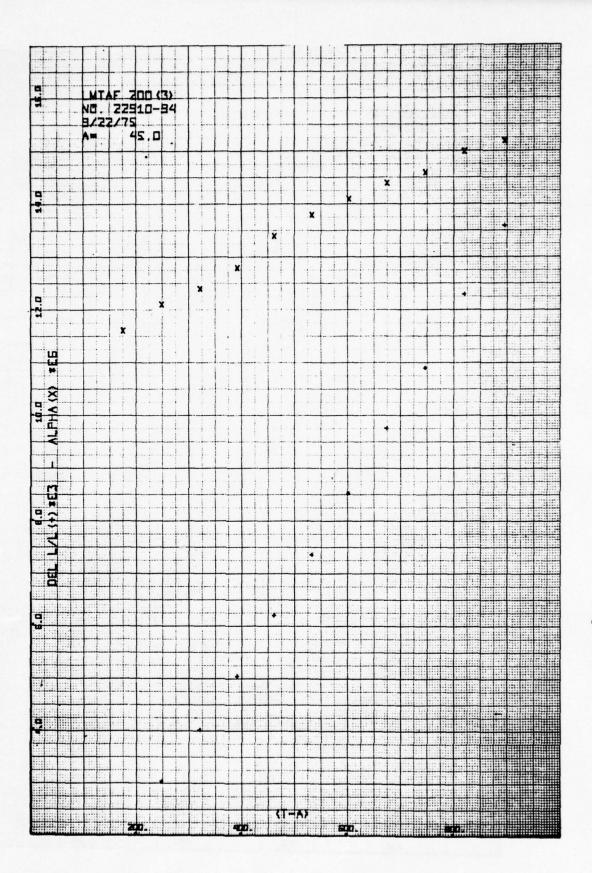


Figure 17 Thermal Expansion (α) vs. Measurement Temperature Minus Ambient (T-A) for Sample LMTF 200(3) with 0.15 Atom Substitution of Al for Fe.

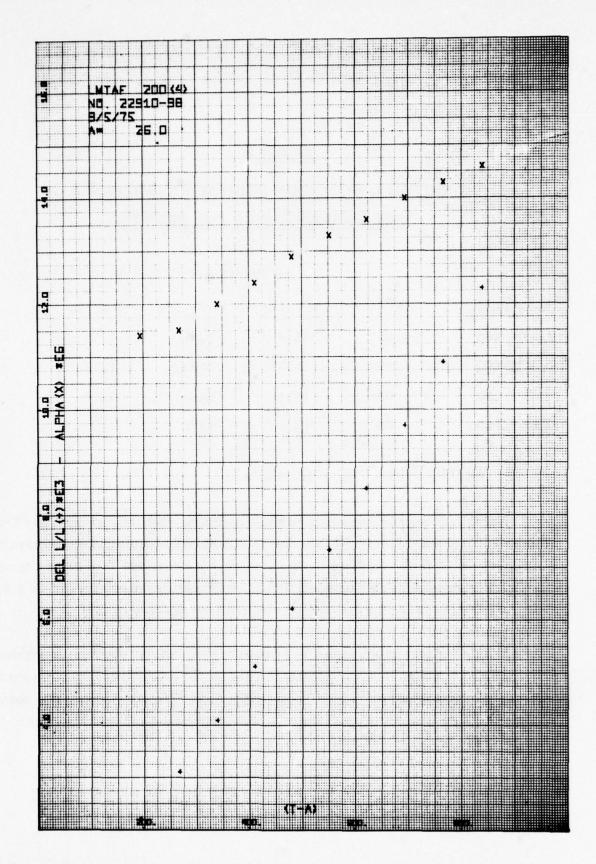


Figure 18 Thermal Expansion (α) vs. Measurement Temperature Minus Ambient (T-A) for Sample LMTF 200(4) with 0.15 Atom Substitution of Al for Fe.

TABLE 2

THERMAL EXPANSION COEFFICIENT AT 1000°C

FOR VARIOUS SPINEL DIELECTRICS

α values in ppm/°C at 1000°C

Designation	<u>*</u>	w* = 0	w = .10	w = .15
LMTF 200	1.00	15.8	15.1	15.0
LMTF 195	.975	15.25	15.0	14.85
LMTF 190	•95	15.1	14.9	14.7
LMTF 180	.90	14.9	14.7	

 * Li .5 + x/2 Mn.1 Ti $_x$ Al $_w$ Fe 2.4-3 x/2- $_y$ O 4

In Fig. 19 we have assembled the expansion coefficient $\overline{\alpha}$ versus (T-A) for the 200 series dielectrics (see Table 2 for composition) as a function of aluminum substitution for iron. One observes a decrease in $\overline{\alpha}$ at any temperature with degree of Al replacement (w). The $\overline{\alpha}$ values at 1000°C (T-A = 980) range from $\overline{\alpha}$ = 15.8 ppm/°C for w = 0 to $\overline{\alpha}$ = 14.6 for w = .25.

A similar plot of $\bar{\alpha}$ versus T-A is shown in Fig. 20 for the 190 dielectrics with w = 0 and w = .15. Smaller values of $\bar{\alpha}$ are found, as one would expect from the reduction in Li-Ti content. However, we do observe some change in slope for the $\bar{\alpha}$ vs. T plot, which, of course, indicates some change in the shape of the expansion curve.

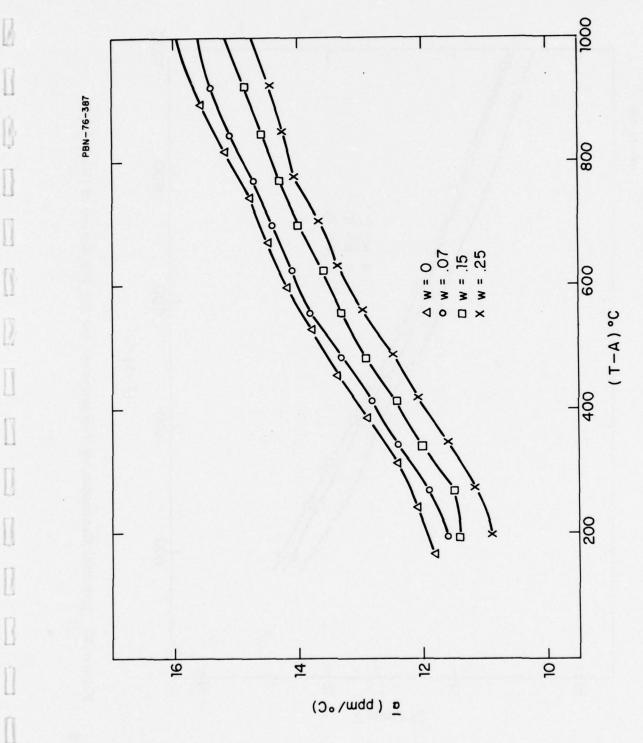


Figure 19 Thermal Expansion vs Temperature for the 200 Series Dielectrics.

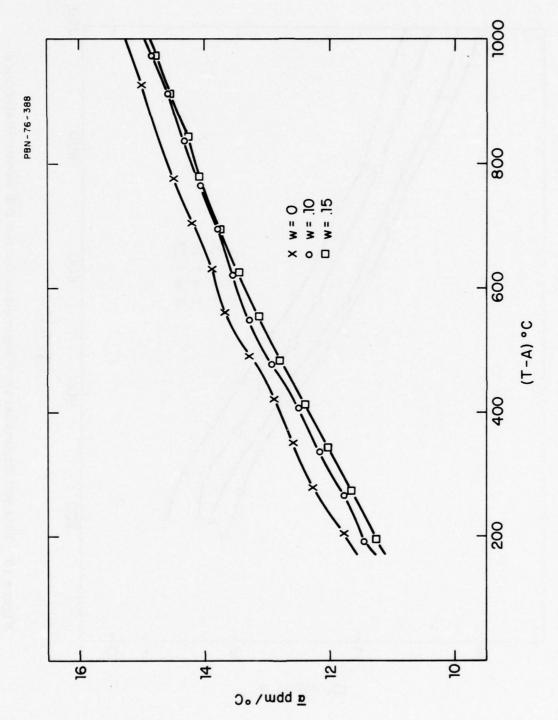


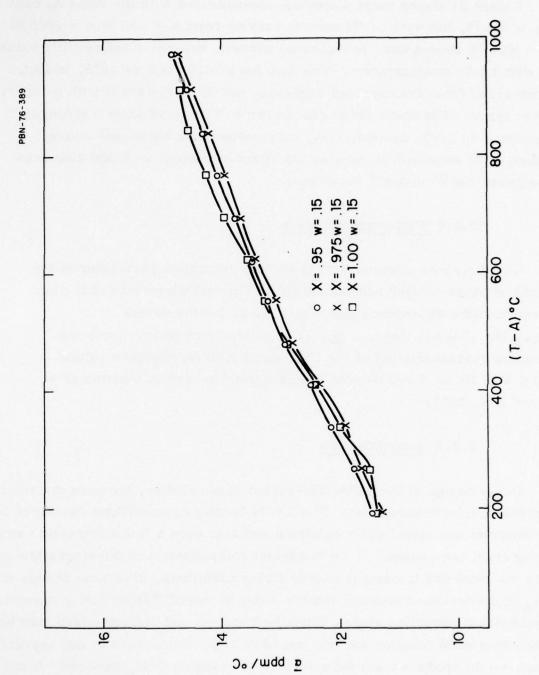
Figure 20 Thermal Expansion vs Temperature for the 190 Series of Dielectrics.

Figure 21 shows three dielectric compositions with the same Al content, w = .15, but with Li-Ti content varying from x = .95 to x = .975 to x = 1.00. It seems that, at higher Al content, expansion shows little variation with Li-Ti concentration. The data for x = .95 and x = .975, in fact, are reversed from the expected sequence, but the variation is within experimental error. The same set of curves for w = 0 would show a stronger dependence on Li-Ti content, i.e., more separation between $\overline{\alpha}$ values. Evidently, Al substitution reduces the effect of content on $\overline{\alpha}$ and also acts to decrease the $\overline{\alpha}$ versus T variation.

2.2.2 Dielectric constant

2.2.3 Magnetization

In the design of the dielectric-loaded phase shifter, the core material is intended to be nonmagnetic. The Li-Ti-ferrite compositions developed for this program are spinel solid solutions and may have a finite magnetic moment arising from two causes: 1) an imperfect compensation of the magnetization in the material due to composition or firing conditions, 2) a local change in $4\pi\,\mathrm{M}_{_{\rm S}}$ at the ferrite-dielectric interface due to interdiffusion during spraying or subsequent annealing steps. Since both ferrite and dielectric are members of the same solid solution series, interdiffusion, if it occurs to any appreciable extent, would produce a graded interface of changing $4\pi\,\mathrm{M}_{_{\rm S}}$ between ~ 0 and 1200 gauss. Evidence to date indicates only minor interdiffusion with annealing. Our main concern at this point is to maintain zero magnetization in the various spinel dielectrics.



Thermal Expansion vs Temperature for Dielectrics with w=0.15 and Variable Li-Ti Content. Figure 21

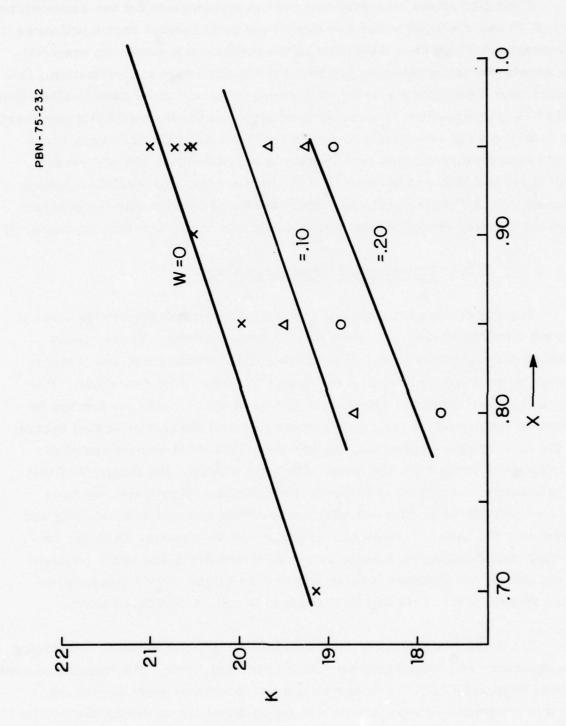


Figure 22 Dielectric Constant at 10 GHz vs Dielectric Composition in the Series $\text{Li.}_{5+\frac{x}{2}}^{\text{Mn.}_{1}} \text{Ti}_{x}^{\text{Al}_{w}}^{\text{Fe}}_{2,4-\frac{3x}{2}} - {}_{w}^{\text{O}}_{4}.$

Figure 23 shows magnetization versus temperature for two compositions (x = 0.70 and x = 0.85) which are beyond the compensated region and three compositions which have been used as the nonmagnetic dielectric material. As shown, the latter three do not have the intended zero magnetization. One should note especially the $4\pi M_S$ vs T curve of the x = 0.95 sample since this LMTF-190 composition is a rather good match in α with the 1200 gauss ferrite and has been used extensively to produce APS phase shifters. We note the Curie temperature for this composition is approximately 160°C, versus 390°C for the 1200 gauss ferrite. This implies that any residual magnetic moment in the "dielectric" would decrease more rapidly with temperature than the ferrite, except for the small initial rise in M_S between 20° and 40°C

2.2.4 Forming and firing large shapes

The dielectric core material for the phase-shifter production must be ground into long shapes with very small cross-sections. These pieces must be strong enough to survive handling and thermal shock and straight enough to mate together well in the assembly before APS deposition. For example, after spraying, the sample is moved about inside the furnace by grasping the dielectric core which extends beyond the ferrite coated section. In the final grinding operation, the sample is also held by sections of dielectric protruding from the ends. Since the strength and integrity of this core material are essential to the success of the APS process, we have devoted considerable time and effort in this first quarter to developing and improving the core. This section summarizes the results. (Another important consideration relating to dielectrics manufacturing is the problem of the cutting and grinding losses, and how we might reduce this to minimize overall cost. This will be discussed in Sec. 4.0 of this report.)

The dielectric powders, produced by conventional ceramic processing are isostatically pressed into bar shapes for final firing. Rectangular shaped rubber bags $1.5 \times 3 \times 16$ inches in internal dimension were purchased for this program. The bags are enclosed in metal forms during the powder filling and isostatic pressing steps to avoid any slight warpage which could

^{*}Trexlar Rubber Co., Ravenna, Ohio

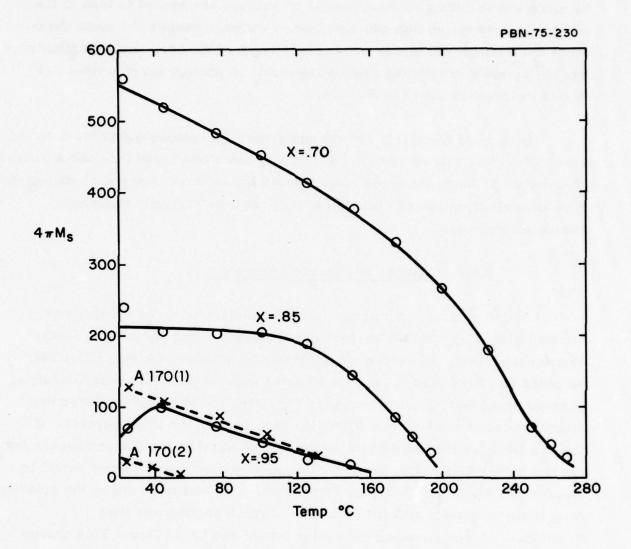


Figure 23 Magnetization versus Temperature for Several Li-Ti-Ferrite Compositions.

be made worse during firing. Special techniques are needed to load in the powder uniformly, as this can also lead to warpage despite the metal form. Since the final ground pieces are the full length of the bar, any distortion in bar shape would drastically reduce the yield. A photograph of a fired bar in this program is shown in Fig. 24.

The bars of dielectric are cut and ground into pieces 0.060 in. \times 0.150 in. in cross section with an .020 \times .020 in. slot along one broad face and a 0.005 in. chamfer at 45° along the edges of the second broad face. Since each dielectric core is made from two of these bars, they must be carefully finished to assure straightness.

2.2.5 Changes in wire slot geometry

A more serious yield problem for the dielectric is the breakage of finished pieces by thermal or mechanical stress during spraying and subsequent annealing. In looking more closely at the slot geometry (Fig. 25), we noted that the 0.020 in. slot depth deing used would weaken the dielectric, perhaps unnecessarily. Referring to Fig. 25a, the slot depth reduces the minimum cross section from 0.060 in. to 0.040 in., or by 33 percent. If the slot were positioned with its major axis parallel to the surface separating the two holves (Fig. 25b), the reduction in minimum cross section would be from 0.060 in. to 0.050 in., or 17 percent. The slot to be cut by the grinding shop in the original 0.060 in. × 0.150 in. cross section was then 0.010 in. × 0.040 in. along the center line of one broad face. This change poses no particular problem or cost increase in machining.

Another advantage of the change from horizontal to vertical long axis for the 0.020 in. × 0.040 in. slot is that exact registration of the two halves is not as critical. With the former (Fig. 25a) a misalignment of a few mils would make it difficult to insert three wires for the polarization switching. A similar misalignment with the second geometry would be far less serious in threading wires down the slot.

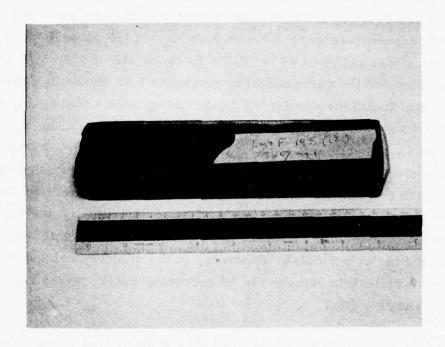


Figure 24 Bar of Li-Ti-Ferrite Dielectric Before Machining.

Since microwave interaction in the loaded phase shifter is fairly complex, it was not obvious a priori whether the change in slot geometry would enhance or degrade the dielectric interaction. A computer program was available which calculates the insertion phase change per inch ($\Delta\Phi/$ in) for this c-band geometry as a function of slot dimensions and location. The ferrite parameters used were ϵ = 19.0 $4\pi \, \mathrm{M_s}$ = 1150 gauss $\mathrm{B_r}$ = 800 gauss. The dielectric was assigned ϵ ! = 19.0. Frequencies of 5.2, 5.5, and 5.7 GHz were calculated for the various slot arrangements as shown in Table 3. With no slot present (solid dielectric) the phase change was -88.18° / in. at 5.5 GHz. With the horizontal slot at center (Fig. 25a) the insertion phase was smaller (-77.29°/in.). A vertical slot of the same dimensions (Fig. 25b) produced more phase change (-81.50°/in.). The present slot configuration has outside slots of $(0.020 \; ext{in.})^2$ and 0.020 imes 0.040 so that with Li-Ti ferrite dielectric (discounting the fact that silicone resin ϵ^{i} = 16 and wire fill the slots) there is an effective phase change smaller than given by either center slot orientation (-69.89° / in. at 5.5 GHz). However, in the standard c-band geometry the volume of dielectric removed is 50 percent greater, so the comparison may not be entirely fair.

The conclusion to be drawn from this study is that there is no penalty, but rather, an advantage in effective dielectric constant using the slot with its major axis vertical. This is important because the effective dielectric constant of the Li-Ti ferrite composite is less than the K-38-garnet composite by about 15 percent(ϵ' eff = 23 versus $\epsilon_{\rm eff}$ = 20) and further reduction due to changes in slot geometry would make one-for-one replacement more difficult. Although this change does not affect magnetic phase shift or microwave insertion loss (and we have used both geometries in this contract), it seems clear that the new geometry (Fig. 25b)has definite advantages for phase shifters produced by APS deposition.

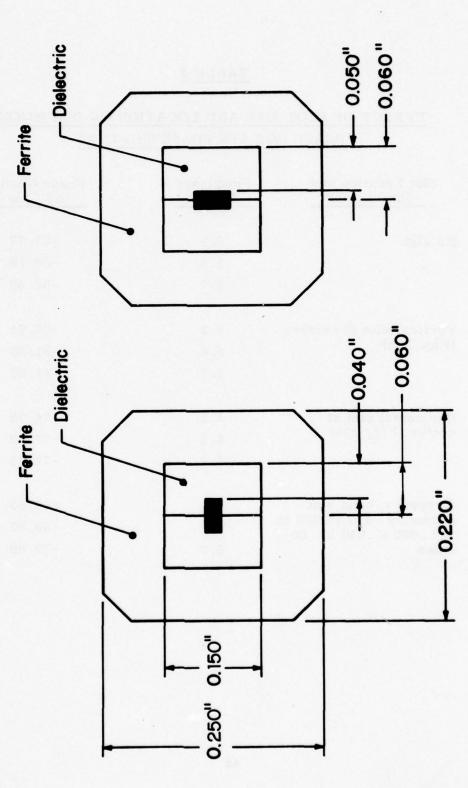


Figure 25 Location of Center Hole in Two-Piece Dielectric.

25a. Early 0.020"×0.040" slot

25. Later 0.020" x 0.040" slot

TABLE 3

EFFECT OF SLOT SIZE AND LOCATION ON INSERTION

PHASE FOR APS PHASE SHIFTERS

Slot Location and Dimensions	Frequency (GHz)	Phase Change ΔΦ°/in
No slot	5. 2	-87.77
	5. 5	-88.18
	5. 7	-88.49
Vertical slot at center	5.2	-80.95
(Fig. 25b)	5.5	-81.50
	5. 7	-81.93
Horizontal slot at	5.2	-76.72
center (Fig. 25a)	5.5	-77. 29
	5.7	-77.25
Present c-band slot	5. 2	-69. 69
geometry $.020 \times .020$ in. and $.020 \times .040$ in. on	5.5	-69.90
sides	5. 7	-70.09

2.3 Design and Construction of Raytheon APS Equipment

2.3.1 Initial design

Design and construction of the plasma-spray equipment was begun soon after the start of the program and was completed before the end of the first quarter. The experimental furnace used in exploratory work was unsuitable for production, both because of inadequate control of furnace temperature and because no more than one sample could be sprayed during a single work day. Furthermore, the original spray station used an inadequate hand crank to provide vertical translation along the rod, which was replaced by a hydraulic assembly in the new design.

The initial configuration is shown in Fig. 26. It shows the overall plan of providing two furnaces so the dielectric rod can be preheated in the upper furnace, maintained at a uniform and constant temperature during spray-coating, and returned to the upper furnace for storage after spraying. The rod would be rotated while moving slowly in a vertical direction during spraying.

The original furnace configuration is shown in Fig. 27. The upper furnace could be physically removed, so that annealing could be performed in a separate location. The upper furnace held both uncoated and ferrite-coated dielectric rods during the spraying operation. Before the start of the spraying workday, the upper furnace was loaded with a batch of dielectric rods and preheated to approximately 800°C. One rod was then placed into a chucking fixture, held from below, and lowered quickly to a position where plasma spraying could begin. Rotation and raising of the rod then proceeded at rates dictated by the plasma-spraying operation. Typical rates used in the ECOM experiments were 0.25 in. to 0.75 in. per minute.

Although the equipment did function well enough to allow the testing out of the double oven idea and the transfer between sprayed and unsprayed samples, there was a need for better temperature control and a better

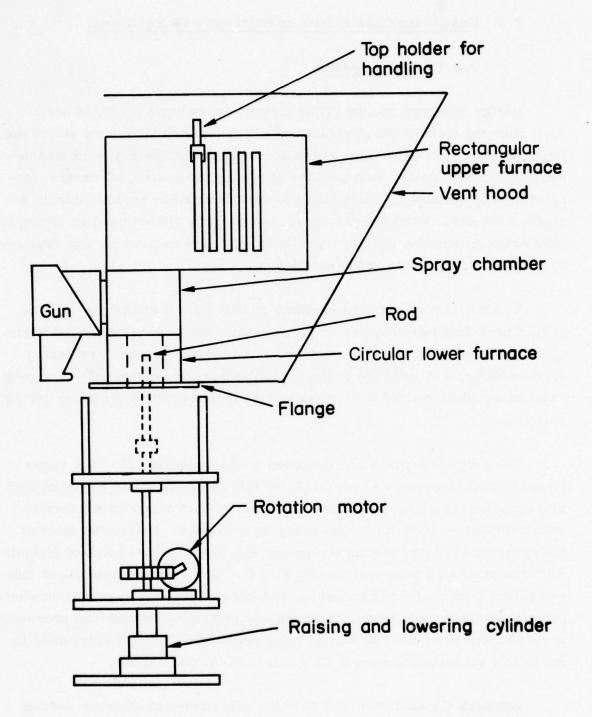


Figure 26 Arc-Plasma-Spray Furnace as Initially Planned.

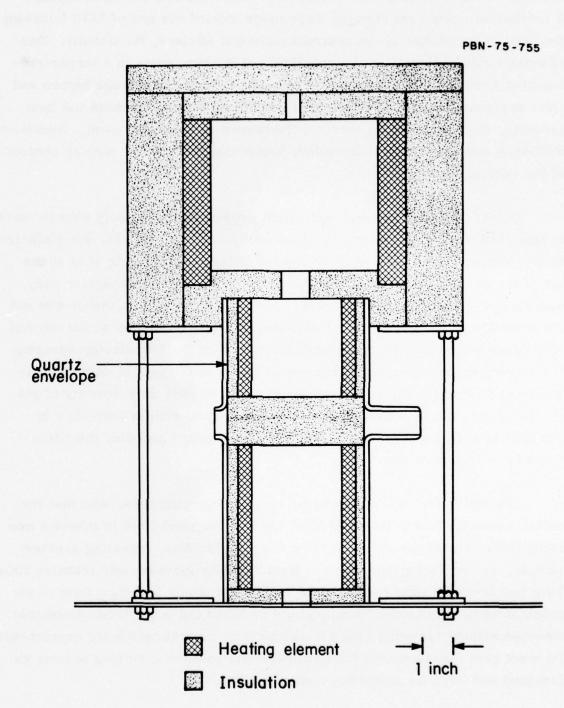


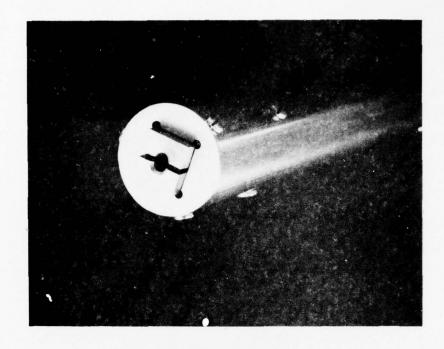
Figure 27 Furnace Arrangement for Arc-Plasma-Spray Unit.

arrangement for holding and rotating the dielectric rod during spraying. A number of equipment changes were made toward the end of 1975 following the visit in November of the contract technical adviser, R. Babbitt. One of these modifications was the rebuilding of the two ovens in a larger rectangular cross-section, making more room for sample storage before and after spraying. A second purpose of rebuilding was to increase the heat capacity, thereby reducing thermal fluctuation in the spray oven. Automatic SCR-type controllers were installed, replacing the manual Variac control of the earlier design.

One of the more serious equipment problems in the early experiments in late 1976 was the unreliability of the early holder design for the dielectric rods. The original holder was a circular hole in the ceramic plug at the top of the sample pedestal which was a close fit for the rectangular rod, making contact the the four corners. At ~ 100 rpm rotation the fit was not close enough to avoid wobble. The dielectric always showed some run-out at the free end and would often work up and out of the hole during spraying A clamping assembly was clearly needed to avoid the problems of holding and transferring the dielectric rods. R. Babbitt gave us a drawing of his clamping assembly, which we had seen and worked with in July 1975 in our preliminary experiments at ECOM. This design provided the basic ideas for our new holder.

The difficulty with Babbitt's holder, for our purposes, was that the entire assembly had to be taken from the furnace each time to insert a new dielectric. Taking the pedestal from the APS furnace, inserting another sample, and replacing the pedestal would greatly increase our transfer time. This fast transfer time had been the rationale for our leaving a hole in the pedestal where the coated sample could be lifted out and a fresh dielectric inserted without lowering furnace temperature or disassembling components. To meet both requirements (fast transfer and positive clamping action) we designed and built the assembly shown in Fig. 28.

The photograph shows two views of the jaw assembly mounted in the rotating pedestal tube. The jaws which grip the dielectric are forced apart



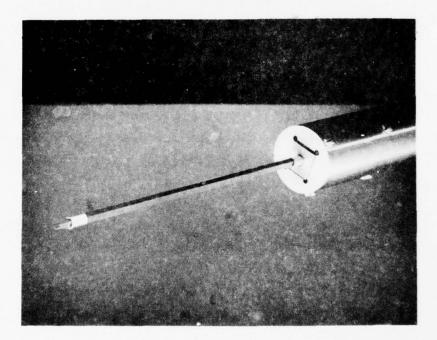


Figure 28 Pedestal Clamp Assembly and Pedestal with Dielectric Rod in Place for APS Deposition.

when moved upward through the square hole in the pedestal cap by pins which ride in the elongate slots. The pins are anchored to the circular cap with a small plate. Below the clamp assembly the connecting rod is hollow. This allows any ceramic pieces to fall through and out of this region. As shown in Fig. 28, the dielectric has metal collars at top and bottom to hold the two halves in position. The jaw clamp shut by a downward spring pull on the connecting rod, as in Babbitt's original design.

2.3.2 Vertical translation equipment

The vertical pedestal motion during APS despoition is controlled by a hydraulic system, which is continuously adjusted in rate to allow precise adjustment in deposit thickness. A schematic diagram of the equipment is shown in Fig. 29. The system allows rapid changes in speed in either direction and at any point in travel. Pressurized air is introduced at the upper left in the diagram and enters a Schraeder three-position, four-way hand valve. The three positions are up, neutral, and down for motion in these same directions. The neutral position stops the motion by venting the air last applied. The direction control valve provides pressurized air in one of the air/oil reservoirs to flow through Scovil valve A or B and then to one side of the hydraulic piston. The flow forces oil out of the opposed piston chamber. This outflow is controlled by one of the two flow valves on the return side of the line. The oil bleeds through the flow valve and into the reservoir, displacing the air at the top, which vents out through the direction control valve.

Table 4 summarizes the different travel modes and control functions for this equipment. For upward piston travel, the control valve air flow is from position 1 to 4, which pushes oil from the lower air oil reservoir through the Scovil B valve (plain arrow indicates unimpeded flow in that direction; crossed arrow indicates valve adjustable flow) and into the lower piston chamber. A controlled rate of rise for the pedestal is achieved by adjusting the slow (Hoke A) or fast (Scovil A) hydraulic valve (see Table 4) which bleeds oil into the upper reservoir and forces air to vent out from



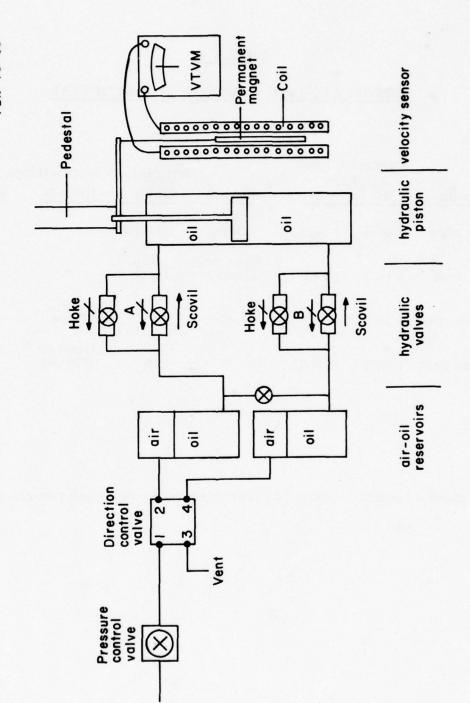


Figure 29 Schematic Diagram of Vertical Translation Equipment.

TABLE 4

MOTION CONTROLS FOR THE APS PEDESTAL

		Control Air F			Hydraulic V	alve Settin	gs
Mode	Motion	In	Out	Hoke A	Scovil A	Hoke B	Scovil B
1	up slow	1 to 4	2 to 3	Adj. Open	Closed	*	*
2	up fast	1 to 4	2 to 3	Open or Closed	Adj. Open	*	*
3	down slow	1 to 2	4 to 3	*	*	Adj. Open	Closed
4	down fast	1 to 2	4 to 3	. *	*	Open or Closed	Adj. Open

Open or closed control function is overridden by the direction control valve.

position 2 to 3 in the direction control valve. Controlled (fast or slow) down-motion can be achieved by changing the air valve position and using the B valves for rate control.

2.3.3 Vertical motion sensor

The monitoring of vertical motion of the pedestal is important for uniform and reproducible coating by the APS process. Since the vertical motion is controlled hydraulically, there is no positive dependence of velocity on dc motor setting and screw pitch as one normally finds with motor control. The hydraulic system, on the other hand, gives a greater flexibility in changing rate or position rapidly. However, one cannot be certain that the hydraulic valves can be reset to reproduce exactly a given velocity. What was needed was a method for sensing the instantaneous pedestal velocity which can be used for final adjustment of the hydraulic control valves. The velocity transducer shown schematically in Fig. 30 has served this purpose very well, giving precise indication between 0.1 in./min. and 100 in./min., which is well in excess of the range of interest.

The sensor works on the simple principle that a permanent magnet of high induction moving axially within a closed coil induces a dc voltage which is proportional to the product of the number of turns of wire per unit length and the number of lines of force generated by the permanent magnet. In effect, the device is simply an electric generator where many turns of wire yield a measurable voltage, even at speeds as low as 0.1 in./min. For velocities the order of one in./min. used in plasma spraying, the output of the velocity transducer is approximately 0.4 millivolt, a voltage easily detected by a standard laboratory voltmeter.

The last of the major changes in equipment design on this contract took place at the end of 1976, between the completion of the confirmatory sample run and the production run. We found that the APS samples were not meeting magnetic property specifications because of distortions in

shape which were evidently occurring during the spray operation. The bowing of samples was rather small, i.e., less than 0.30 in. in the six-inch length, but this amount was enough to cause significant changes in ferrite wall thickness and in magnetic properties, particularly a reduction in phase shift. It was decided that one contributing factor might be nonconcentricity or wobble in the pedestal assembly relative to the plasma gun.

We theorized that radial nonuniformities in the deposit pattern would produce differential temperatures and differential strains, which could produce the warping observed. This situation could be brought under control by a more exactly aligned system.

The translation equipment was therefore redesigned and rebuilt to improve its reliability for the APS process. A photograph of the redesigned pedestal tube assembly is shown in Fig. 30. The tube and rotational drive motor move up and down on the rectangular block (b), which is guided by linear bearings within the block and the two vertical guide rods (g). The end of the pedestal tube (p) is made to rotate on axis by screw adjustments on two metal disks at the base of the tube having a 0.5 in. steel ball captured in retaining slots between the disks. This pedestal assembly, which has leveling screws at the base, sits on a steel plate, which is attached to metal rods suspended from another plate. This second steel plate underlies the plasma-spray furnace and the upper holding oven. Such an elaborate arrangement of metal support plates and interconnected equipment (shown schematically in Fig. 31) was designed so that the pedestal assembly and spray and holding ovens are interconnected and cannot move independently.

At this point there was still the possibility that the stubstrate would wobble at the free end. To avoid this problem, an upper idler bearing (see dashed lines) was built which could be used to capture the free end of the substrate. In this scheme a graphite part would replace the upper metal clip and provide a conical tip for rotation within the bearing tube.

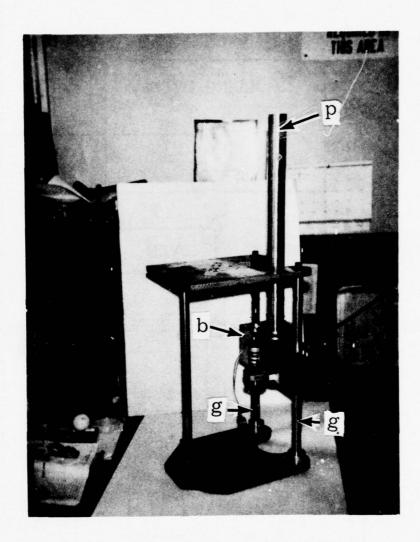


Figure 30 Pedestal Tube Assembly for Arc-Plasma Spraying.

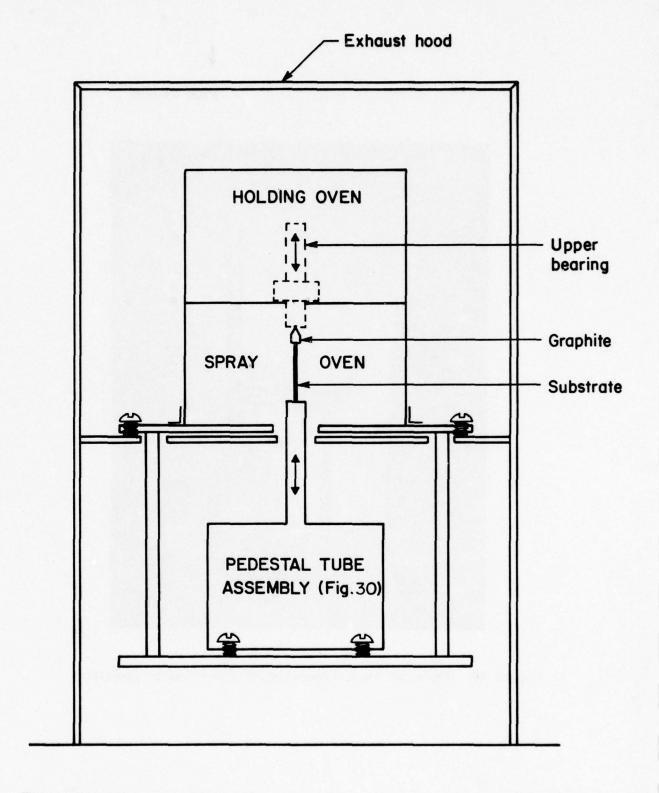


Figure 31 Diagram of Metal Supporting Plates and Interconnected Equipment.

Fortunately, it was not necessary to use the upper bearing to avoid substrate wobble. The improved pedestal assembly (Fig. 30) was, in fact, sufficient. The objection to the use of the bearing is that it would have to be removed after each run for sample transfer into the holding oven. The bearing would be hot (600°C) and difficult to maneuver.

2.4 Experimental APS Runs

2.4.1 Initial experiments at USAECOM Labs

In July 1975, just after the start of the contract, a four-day visit was arranged at USAECOM Laboratories to conduct APS runs using Army equipment with the guidance of R. W. Babbitt. Machined substrates and the Liferrite spray-dried ferrite powder to be used in these experiments were produced at Raytheon before the trip. Our purpose was to learn as much as possible about APS procedures at ECOM and to test out our starting materials with their equipment.

A summary sheet of the experimental conditions used are shown in Table 5. In these experiments a mixture of argon and helium (~10:1 ratio, see col. 4) was used whereas in subsequent work only argon was used. The oxygen carrier gas flow rates in these experiments were also higher than flow rates used in subsequent work.

Some limited studies of hysteresis properties and microwave evaluation were made on two of the samples from this preliminary run. The results are summarized in Table 6. Sample R13, which was 2.5 in. after machining (originally full-length samples were made) gave 57.6° phase shift $(\Delta \phi)$ per inch with one turn 15 amp drive. This corresponds to 296° for a full-size toroid. Insertion loss would be > 2 dB if this sample were full size.

The second sample, No. R7, had somewhat better properties $(\Delta \phi) = 74.4^{\circ}/\text{ in.}$ and insertion loss (I.L.) of 0.16 dB/in.) giving $\Delta \phi > 340^{\circ}$ and I.L. < 1 dB if the toroid were full size.

2.4.2 Early APS experiments at Raytheon

Following the initial experiments at Fort Monmouth, we began APS runs at Raytheon using equipment modified in light of the experience at USAECOM Laboratories. During the period of November 1975 through

TABLE 5

APS EXPERIMENTAL RUNS MADE AT ECOM LABORATORIES

Anneal (°C)	0911					1020	•		Aborted		•		1000	•	
Spray Time (min.)	13	=	=	8 1/2	8 1/2	6	16 1/2	01	Run	9 1/2	10 1/2	11 1/2	12	15	. 13
oven (C)	620-680	650-750	750-800	750-800	750-780	092-002	740	092-002	700-720	720	720	710	200	710-675	700-725
Spray Distante (in.)	2	2	2	2	2	2	2	1 3/4	13/4	1 3/4	2	2	2	2	2
Powder Feed (cu.ft./hr.)	65	. 75	75	75	001	75	55	80	08	78	75	78	02	02	02
Oxygen Carrier Gas (cu, ft, /hr.)	75	08	8	8	100	80	09	80	80	85	85	85	80	08	80
Arc Current (amps)	320	350	350	350	320	350	350	350	350	350	350	350	320	320	320
Arc Gas (cu. ft/hr.)	20/59	70/07	70/07	20/08	80/20	75/07	75/07	75/07	75/07	75/07	75/07	75/07	78/07	78/07	78/07
Arc (cu. ft	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He	Ar/He
Dielectric Substrate	LMTF 190 (3, 4)	LMTAF 200 (3)	LMTAF 180 (3)	LMTF 200 (1)	LMTF 190 (3, 4)	LMTAF 200 (3)	LMTF 190 (3, 4)	LMTF 190 (3, 4)	LMTAF 180 (3)	LMTAF 180 (3)	LMTF 200 (1)	LMTAF 200 (3)	LMTF 190 (3, 4)	LMTAF 180 (3)	LMTAF 180 (3)
Ferrite Powder	LMTF 53	(B_										>	Ampex 1202	Ampex 1202	LMTF 53
Run	1	2	3	4	2	9	1	∞	6	10	=	12	13	14	15
Date	7128175						7129175							7/30/75	

TABLE 6

DATA ON ENGINEERING TEST SAMPLES

(5.5GHz)

Test S Location	Sample No.	Length (in.)	Drive Turns	Drive Current	H _c (Oe)	4πM _r (gauss)	Δφ (deg.)	In. Loss	Ret. Loss
Anneale	d 1000°C	, 1 hour	r in air						
Raytheon	R13	~2.7	2	12	2.9	790		aled follo	wing APS
ECOM	R13	2.50	1	6	1.96	380	58.5	1000°C)	
ECOM	R13	2.50	1	8	2.24	497	99		
ECOM	R13	2.50	2	5	2.51	600			
ECOM	R13	2.50	1	10			117		
ECOM	R13	2.50	1	15			144	. 75	15
ECOM	R13	2.50	1	20			149		
Raytheon	R13	2.50	1	6	1.94	206			
Raytheon	R13	2.50	1	8	2.68	446			
Raytheon	R13	2.50	1	15	2.99	614			
Raytheon	R13	2.50	2	6	2.88	639			
Raytheon	R13	2.50	2	8	3.05	712			
Raytheon	R13	2.50	2	15	3.09	749			
Annealed	1044°C	, 1 hour	in air						
Raytheon	R13	2.50	2	15	2.53	820			
Raytheon	R13	2.50	1	15			186	.4dB	19
Raytheon	R7	2.36	2	15	7.62	574	(APS	sprayed,	no anneal)
Anneale	d 1044°	C, 1 hou	r in air						
Raytheon	R7	2.36	2	15	2.95	649			
Raytheon	R7	2.36	1	15			140	1.0	15

Test performed at ambient temperature (21°C) in air on arc-plasma-sprayed samples of Li-Mn-Ti ferrite deposited on dielectric type LMTF-190(34) shaped into c-band geometry as described in text.

June 1976 seven sets of engineering samples were tested and reported on. Appendix III is the APS log of all the samples sprayed at Raytheon during this contract. Samples for the engineering tests represent those taken from the first 100 sprayed samples. Progress during this time was slow. Delays were primarily due to equipment problems associated with the changes from experimental apparatus to equipment compatible with the manufacturing rate required by the program. We found that steps leading from the design of apparatus capable of spraying a few samples per day to several per hour are not simple steps. We did encounter serious problems with sample cracking in the third and fourth engineering sample deliveries. In retrospect, the cracking problem was more likely attributable to the ferrite powder than to spray conditions.

Microwave properties on the toroidal engineering samples (sixth and seventh series) generally gave low insertion loss (~1 dB) but the saturation phase shift was typically 280° to 300°, about 15 percent below the contract requirement.

2.4.3 Confirmatory sample production

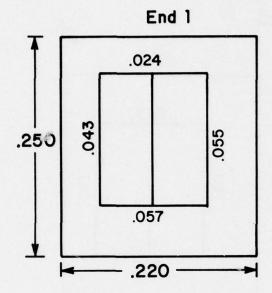
The contract schedule had called for the delivery of 20 confirmatory samples (full-size phase shifters) and a report at the end of October 1976. During the summer a large number of samples were sprayed in preparation for this testing. As the test results accumulated on these full-size phase-shifter samples, it became evident that the samples would not pass the confirmatory test because of a low $B_{\rm r}$ and therefore a phase shift below the required 340°. The $B_{\rm r}$ values were not only low, but showed considerable variation from one sample to the next, with no apparent relation to dielectric composition or spray conditions. We decided to section two of the full-size phase shifters which had low $B_{\rm r}$. The samples were APS 170 with $B_{\rm r}$ = 508 gauss and APS 174 with $B_{\rm r}$ = 565 gauss. They had been sprayed during a session when other samples having good hysteresis loop and microwave properties were produced.

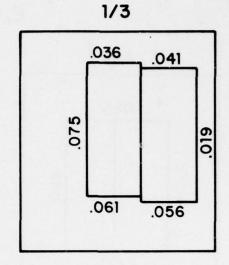
Each of the samples showed reasonably uniform ferrite walls at the exposed ends (see end 1 and end 2 in Figs. 32 and 33). The 5.145-in. samples were cut into three equal segments, which exposed two surfaces at the one-third distance (see 1/3, Fig. 32) and two surfaces at two-thirds the original length. For sample APS 170 the two dielectric halves showed 0.005-in. displacement at the on-third position and a severe nonuniformity in wall thickness. At the two-thirds position the wall was still nonuniform, the thin side remaining the same. The entire center segment evidently has one narrow and one thick wall, a condition which would be expected to produce a very low B_r. The final segment of APS, between the two-thirds location and end 2, has a nonuniform wall. The dielectric halves were still displaced 0.005 in. but were reasonably uniform at the ends. A similar dissection of sample APS 174 (Fig. 33) showed similar wall nonuniformities, although not as extreme as APS 170.

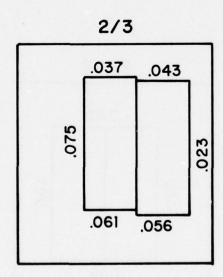
If we examine the machining process, it will be evident why the ferrite walls appear uniform at the ends and can still be a very nonuniform in the center. The machinist keys the grinding away of excess ferrite to the extreme ends of the sample where the bare dielectric rod extends beyond the ferrite coating. At the ends of the rod, then, assuming the machinist does his job, the ferrite coating around the dielectric is a uniform 0.050 in. These are the regions we see in cross section when the phaser is cut to its final length. Only destructive sectioning of the element would reveal the wall uniformities in the center regions prior to the development of X-ray transmission techniques.

It was evident from these findings that we could not meet the target date for the sample delivery. We asked for and received permission to delay the delivery of the confirmatory samples until January 1977. The last of the APS samples used in the confirmatory run were sprayed in early December. By this time we had obtained a new powder, LMTF 475(G5), (see Table 7) with a higher $4\pi M_S$ which, with allowance for a lower APS density, gave $4\pi M_S$ = 1200 gauss and a more favorable B_r and phase shift.

Sample APS 170 $B_r = 508$







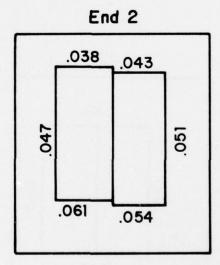
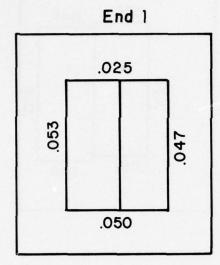
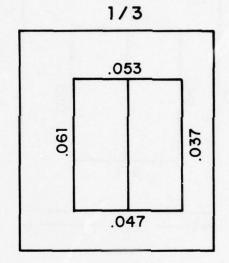
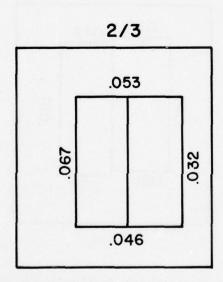


Figure 32 Cross Sections of Plasma Sprayed and Machined Phase Shifters. Wall thickness in inches as indicated

Sample APS 174 $B_r = 565$







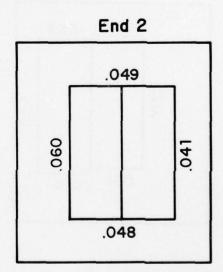


Figure 33 Cross Sections of Plasma Sprayed and Machined Phase Shifters. Wall thickness in inches as indicated

TABLE 7

CONFIRMATORY SAMPLE TEST RESULTS (MIL-STD 831, Para, 5.6.10.1.4)

	1	H, dB								1.2				.3								
	+ 85°C	(c)								3829				3755								
	+	1000																				
	1	L.								1.4 315				1.0 318								
5 GHz	- 30°C	(o) I.								3687				3430								
at 5, 4	1	(0)								477 36				454 34								
RF Properties at 5,45 GHz	1	1. L. dB			0	4	1	1.88						09.0	1.38	1. 20	0.58	09 '0				
RF Pro					1.0	1.4	16 0.7			1.1												
	25°C	0 (0) TO(0)			3732	3711	3646	3715		3760				3636	3668	3720	3658	3671				
		D & D			346	355	366	400		371				383	380	416	410	423				
		(in.)	5. 145	5, 145	5, 145	5, 114	5.115	5, 145	5, 145	5, 145	5.145	5, 145	5, 145	5, 145	5, 145	5, 145	5, 145	5, 145	5, 145	5, 145	5, 145	5.145
		Δ B _r (%)	± 6.6	± 7.2							1 9.5	1 8.1	t 8.3	t 7.7	1 10.1	£ 5.6	+ 6.4	1 7.2				
	ان	Br (g)	591	918							665	682	099	999	099	711	730	804				
ωl	+ 85°C	r (0e)	2.60	2.55							2. 22	2.28	2.05	2.47	1.64	2.43	2. 12	1.22				
opertie		3 (g) 1	675	665							805	808	622	111	808	962	830	929				
Loop Properties	- 30°C	H _c (Oe) B _r (g) H _c (Oe) B _r (g)	3.87	3.60							3, 30	3, 33	2.80	3. 52	2.15	3.35	3.43	1.80				
					989	989	715	725	089	733									171	792	739	748
	25℃	B (g)																				
		Ис (Ое)	3.13	3.02	2.84	3. 12	2.32	2.75	2.45	2.68	2.70	2.84	2.43	2.84	1.85	2.85	2.53	1.46	2.39	2.67	2.91	2, 99
		Temp.	*	×							×	×	×	×	×	×	×	*				
		W.G. Phase Shift			×	*	*	×		×				×	×	×	*	×				
		A PS No.	191	162	163	176	225	234	238	241	244	264	270	274	277	279	281	282	286	289	291	293

Some of the samples sprayed with this new powder were reported on in the confirmatory sample report.

Tables 7 and 8 summarize the test results on the confirmatory samples. The number on the last of the samples, APS 293, indicates that almost 200 boules had been sprayed during the summer and fall of 1976 to produce the necessary 20 samples. In addition to the machining and testing on about half of these samples, a nondestructive technique (X-ray fluoroscopy) was developed to assess sample straightness after firing (see Appendix II) and to devise techniques to avoid this problem.

Table 8 shows H_c and B_r measured at 25°C on all 20 full-size phase shifters. Typical values of H_c are 2.5 - 3 Oe with B_r ranging from 650 to 870 gauss. The change in B_r between -30° and +85°C varied between ± 6.5 and ± 10.1 percent about a mean value. We speculate that residual strains are a contributing factor to the variations in B_r . These same compositions fired conventionally show a temperature variation of ± 9.2 percent over the same interval. The smaller percentage change in B_r for samples such as APS 279 indicates that the postulated stress effects are reducing temperature dependencies without sacrificing B_r or phase shift.

The microwave phase shift on ten of the confirmatory samples is shown in column 12 in Table 7. All of the samples exceed the required 340°. Insertion loss at 25°C is < 1 dB on about one-half of these samples. Insertion phase shows a rather disappointing variation from 3640 to 3760, a spread in phase which is typical of the present garnet-K-38 device. The mean square deviation in the series is 41°.

Table 8 shows the temperature variation in $\rm H_{c}$ and $\rm B_{r}$ from -30° to +85°C.

ECOM laboratories remeasured the ten confirmatory samples mounted in waveguides which were sent with the test results (Table 7). A comparison of the measurement of saturation phase shift ($\Delta \phi$ °) and insertion loss (I.L.)

TABLE 8

		(%)	1.1	9.	.2	.5	-	.3			9	4	,
		AB	L	+ 6.6	± 7.2	± 9.5	± 8.1	± 8.3	± 7.7	110.1	± 5.6	± 6.4	
			85°C	591	576	665	682	099	999	099	111	730	
			50°C	630	611	722	736	710	705	719	749	169	
		(s)	25°C	649	636	754	191	734	733	753	770	789	
THRE		B (gauss)	000	629	651	781	787	754	754	781	787	812	
MPERA	4)		-10°C	099	655	787	793	763	763	792	787	815	
S VS TF	6. 10. 1.		-30°C	675	999	808	808	179	777	808	962	830	
FR TIES	ara. 5.		' 1										
HYSTERESIS LOOP PROPERTIES "& TEMPERATHRE	(MIL-STD 831, Para. 5.6.10.1.4)		85°C	2.60	2.55	2.22	2.28	2.05	2.47	1.64	2.43	2.12	
ESIS LO	(MIL-ST		20°C	2.84	2.75	2.47	2.54	2.21	2.73	1.79	2.84	2.31	1
HYSTER			25°C	3.13	3.02	2.70	2.84	2.43	2.84	1.85	2.85	2.53	,
		e l											
		H _c (0	30°C -10°C 0°C	3		2.	2.	5.	3,	2,	3,	2	
			-100	3.52	3.5	3.0	3.24	2.6	3.36	2.13	3.18	2.8	
			-30°C	3.87	3.60	3.30	3.33	2.80	3.52	2.15	3.35	3.43	
			Foroid No.	161	162	244	264	270	274	277	279	281	

at 5.5 GHz and 25°C made at Raytheon and at the ECOM Laboratories is shown in Table 9. The differences between the Raytheon and ECOM results were negligible.

TABLE 9

Comparison of Microwave Data on Confirmatory Samples

		neon Results		Results
Sample No.	$\Delta \phi$ (deg.)	I.L. (dB)	$\Delta \phi$ (deg.)	I.L. (dB)
163	346	1.0	340	0.9
176	355	1.4	360	1.3
225	366	0.7	370	1.0
234	400	1.88	390	2.0*
241	371	1.1	400	1.2
274	383	0.60	400	0.9
277	380	1.38	400	0.9
279	416	1.20	420	1.5
281	410	0.58	425	0.7
282	423	0.60	425	0.6

^{*} Reduced to 1.3 dB after additional one-hour anneal at 970°C.

2.4.4 Pilot production of 200 APS samples

After delivery of the 20 confirmatory samples in January, we began construction of the support elements for the ovens and spray equipment, as described in Section 2.3. The objective was to minimize the chance for sample wobble, which we believed responsible for the problems of sample bowing that caused the slip in delivery schedule. In addition to these changes, a further alteration was made in the holder geometry. The jaws (Fig. 28) which clamped the bare dielectric rod during spraying were replaced with a large tapered hole ~ 0.7 in. in diameter which would accept a graphite plug rather than the bare dielectric. The dielectric was forced into a hole machined in the center of the graphite plug. This change helped to reduce wobble substantially and also made it easier to transfer and store sprayed samples.

After making these changes in the equipment, a series of tests were run to prove out the new equipment. Approximately 50 samples were sprayed (APS 294 through APS 340) to test out the production process. A supply of spray-dried ferrite powder with the nominal composition $\text{Li}_{.735}\text{Mn}_{.10}\text{Ti}_{.475}$ $\text{Fe}_{1.69}\text{O}_4$ was ordered from Raytheon SMDO, and several bars of dielectric with composition $\text{Li}_{1.0}\text{Mn}_{.10}\text{Ti}_{1.0}\text{Al}_{.07}\text{Fe}_{.83}\text{O}_4$ were fired and sent for machining. The wire slot on these pieces was machined with the long (0.040 in.) dimension parallel to the join between halves (Fig. 25b).

Permission to proceed with the pilot production run was received informally (by telephone) on April 4 and officially on May 7. The samples were to be sprayed at a rate of five per hour and, after annealing and machining to the final phase-shifter dimensions, to pass certain microwave tests. The production run was divided into five batches of 40 units. Of the 40 phase shifters, a government representative (DECASPRO) selected 20 which would be subject to hysteresis loop and microwave testing under government supervision.

2.4.4.1 First production batch

Table 10 shows the APS phase-shifter units, the X indicating random selections of the 20 whose hysteresis loops were tested. From these 20 samples, 10 were selected for microwave testing (Table 11), and from these 10 two were selected for microwave testing versus temperature (Table 12).

In general, the results on phase-shifter properties were good. Phase shift at 5.45 GHz was between 390° and 420°, which was well above the >340° requirement. Insertion loss was >1 dB for about half of the samples tested. Insertion phase, however, was disappointing in terms of contract goals (s.d. = ± 15 °) showing variations from the mean of ± 113 to -131. For a total insertion length of 3700° this amounts to ± 3.5 percent variation of phase.

APS PHASE SHIFTER ELEMENTS SELECTION

BATCH NO. 1

Units		lections		Units		elections	0
Received	20	10	2	Received	<u>20</u>	10	2
303				353	X	X	
308	X			354	X	X	
317				358	X	X	
318				360			
320				363			
321	X			364			
325	X	X	X	367	X		
327				373	X	X	
328				374			
330				375	X		
331	X	X		378	X		
332	X			379	X		
335				380			
337	X			38 2			
338				3 83	X	X	
343				384			
345	X			385			
346	X	X	X	386	X	X	
348				387			
349	x			388	X	X	
351							

TABLE 11

APS PHASE SHIFTERS BATCH NO. 1

MICROWAVE MEASUREMENTS - ROOM TEMPERATURE

Serial No. 5.2 5.45 5.2 5.45 5.45 5.45 5.45 5.45 5.45 5.45 5.45 5.45 5.45 5.7 5.45 5.45 5.7 5.45 5.7 5.45 5.7 5.45 5.7 5.7 5.45 5.7 5.4 5.7 5.4 5.7 5.7 5.2 5.7 5.7 5.8 5.10 5.7 5.7 5.8 5.10 5.7 5.7 5.8 5.10 5.7 5.7 5.8 5.10 5.7 5.7 5.8 7.10 5.7 5.7 5.8 7.10 5.7 5.7 5.8 7.10 7.2 <			Δφ (°)			I.L. (dB)			φ IN (c)	
418 416 0.86 0.78 1.48 -125 -128 411 409 408 1.24 1.16 1.64 - 97 - 98 412 410 409 1.86 1.78 2.71 -131 - 131 400 397 397 0.42 0.67 0.64 REF REF 409 408 1.02 1.06 1.42 + 1 + 6 409 408 0.50 0.35 0.60 - 43 - 44 408 407 0.84 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 - 116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 - 119 - 118	ial No.	5.2	5.45	5.7	5.2	5.45	5.7	5.2	5.45	5,7
411 409 408 1.24 1.16 1.64 - 97 - 98 412 410 409 1.86 1.78 2.71 -131 -131 400 397 9.42 0.67 0.64 REF REF 389 390 388 1.02 1.06 1.42 + 1 + 6 409 408 0.50 0.35 0.60 - 43 - 44 408 407 0.68 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	325	418	418	416	0.86	0.78	1.48	-125	-128	-135
412 410 409 1.86 1.78 2.71 -131 -131 400 397 397 0.42 0.67 0.64 REF REF 389 390 388 1.02 1.06 1.42 + 1 + 6 409 408 408 0.50 0.35 0.60 - 43 - 44 408 407 0.68 0.76 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	331	411	409	408	1.24	1.16	1.64	- 97	- 98	-106
400 397 397 0.42 0.67 0.64 REF REF 389 390 388 1.02 1.06 1.42 + 1 + 6 409 408 0.50 0.35 0.60 - 43 - 44 408 407 0.84 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	346	412	410	409	1.86	1.78	2.71	-131	-131	-137
389 388 1.02 1.06 1.42 + 1 + 6 409 408 0.50 0.35 0.60 - 43 - 44 408 407 0.84 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	353	400	397	397	0.42	0.67	0.64	REF	REF	REF
409 408 408 0.50 0.35 0.60 - 43 - 44 408 407 0.84 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	354	389	390	388	1.02	1.06	1.42	+	9 +	+ 7
408 407 0.84 0.76 1.60 +120 +113 408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	358	409	408	408	0.50	0.35	09.0	- 43	- 44	- 53
408 408 407 0.68 0.52 1.36 -113 -116 404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	373	408	407	407	0.84	0.76	1.60	+120	+113	66 +
404 403 401 1.00 0.93 1.98 - 95 - 96 422 421 420 0.70 0.64 1.52 -119 -118	383	408	408	407	0.68	0.52	1.36	-113	-116	-124
422 421 420 0.70 0.64 1.52 -119 -118	386	404	403	401	1.00	0.93	1.98	- 95	96 -	-103
	388	422	421	420	0.70	0.64	1.52	-119	-118	-126

TABLE 12

APS PHASE SHIFTER ELEMENTS BATCH NO. 1

TEMPERATURE MEASUREMENTS

Serial No. APS-325

			5.2 GHz			5.45 GHz			5.7 GHz	
		-30€	25°	+85°	-30°	25°	+85°	-30,	25.	+85°
	I.L. (Db)	4. 18	0.86	1.80	1.56	0.78	1,64	1.36	1.48	2.06
	Δφ (٠)	513	418	335	535	418	337	250	416	340
76	φ IN (°)	+124	REF	- 58	+102	REF	- 56	+ 97	REF	- 54
	Serial No. APS-346	PS-346								

		5.2 GIIz			5.45 GHz			5.7 GHz	
	-30°	25°	+85°	- 30°	25°	+82°	-30°	25°	+82°
I.L. (dB)	1.85	1.86	3.0	1.24	1.78	2.88	1.12	2,71	3.44
(₀) ♦∇	556	412	333	567	410	335	576	409	337
(°) VI ¢	+105	REF	-45	+ 91	REF	- 42	+ 83	REF	- 41

2.4.4.2 Second production batch

Table 13 indicates the 20 selected units and the 10 samples selected from this for microwave testing. The plasma log table (Appendix III) describes the spray conditions used for the 52 units (APS 439-388 inclusive) which were sprayed for batch No. 2.

Microwave data Table 14 shows phase shift is again high 372° - 414° and insertion loss meets the goal of <1 dB for half the samples. The very high loss for APS 424 is probably due to incomplete oxidation during the 1015°C two hour anneal. Insertion loss shows somewhat less of a spread (±80°) than that observed for batch No. 1. The temperature variation (Table 15) is typically +150° at -30°C and -30° at +85°C relative to the room temperature value.

Of the total 52 samples sprayed, twelve were lost in processing and testing. Five of these were broken during machining, two broke or chipped during annealing and the remaining five were rejected for cracks or bowing as revealed by X-ray fluoroscopy testing. The latter five were not machined.

2.4.4.3 Third production batch

The third batch APS 439 through 500 comprised 62 sprayed samples. The selection of 20 test samples was made on June 8 (Table 16) and from these 10 were given the required microwave testing (Table 17). We note that the phase shift is lower than the average from previous batches, most likely due to problems incracking of the ferrite coating. Insertion loss was again > 1 dB for half of the samples tested but losses were considerably higher in the cases remaining. Insertion phase variations shows fluctuations of \pm 108 to -114 relative to a mean value.

The temperature variation of insertion phase (Table 18) in APS 442 (+229° to -117°) is very large relative to APS 476 (+88 to -42°) between -30° and ±85°C. These two samples were sprayed with the same ferrite

APS PHASE SHIFTER ELEMENTS SELECTION

BATCH NO. 2

Units Received	<u>20</u>	Selections 10	2	Units Received	<u>20</u>	Selections 10	2
344				413	· in the		
347	X			415	X		
390	X	X		417	X		
391				422			
392				423	X		
393				424	X	X	
394	X	X		426	X		
395				427			
396				428	X	X	
397	X	X		429			
398				430	X		
399	X	X		431	X	X	
400				432			
401				433	X		
405				434			
406				435	X		
407				436			
408	X	X		437	X		
411	X	X	X	438			
412	X	X	X	439	X		

TABLE 14

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APS PHASE SHIFTERS BATCH NO. 2

MICROWAVE MEASUREMENTS - ROOM TEMPERATURE

	5.7	+	98+	+53	+20	-19	REF	-20	-80	+21	2
φ _{IN} (°)	5,45	+	+82	674	449	-17	REF	-20	-81	+22	e
	5.2	- 1	180	446	446	-19	REF	-20	-83	+19	9 -
	5.7	1.94	0.84	0.58	0.46	1.40	1.16	1.56	1	1.14	2.08
I. L. (dB)	5.45	1.68	0.96	0.72	0.45	0.63	0.68	1.14	7.0 dB	1.00	1.54
	5.2	1.72	0.76	99.0	0.56	0.40	0.40	0.76	!	96.0	1.40
	5.7	403	369	395	395	396	414	410	399	377	386
(°) \$\Delta \Delta \Delta	5.45	404	372	396	396	397	414	410	399	379	389
	5.2	405	374	397	400	398	415	411	402	380	390
	No.										
	Serial No.	390	394	397	399	408	411	412	424	428	431

TABLE 15

APS PHASE SHIFTER ELEMENTS BATCH NO. 2

TEMPERATURE MEASUREMENTS

No. APS 411		
APS	11	
APS	4	
No.	APS	The state of the s
Serial	No.	

		5.2 GHz			5.45 GHz			5.7 GHz	
2	-30°	25°	+85°	-30°	25°	+85.	-30	25°	+82.
I.L. (dB)	2.0	0.40	09.0	0.66	0.68	92.0	1.36	1.16	1.00
(°) ∳∇	554	415	334	535	414	337	523	414	337
φ IN (°)	+154	REF	-63	+124	REF	-57	+104	REF	-50

Serial No. APS 412

		5.2 GHz		5	5.45 GHz			5.7 GHz	
	-30°	25°	+82°	-30°	25°	+82.	-30	25°	+82.
I.L. (dB)	5.12	0.76	0.64	1.16	1.14	0.82	1.04	1.56	1.20
(°) ∳∆	538	411	330	525	410	333	512	410	334
φ IN (°)	+167	REF	-50	+134	REF	-47	+114	REF	-42

APS PHASE SHIFTER ELEMENTS SELECTION

BATCH NO. 3

Units Received	20	Selections 10	2	Units Received	20	Selections 10	2
440				463	X		
442	X	X	X	464			
444	X	X		466	X		
445				467			
446				468			
447	X	X		469	X		
449				471			
450				472	X		
451	X	X		475			
452	X	X		476	X	X	X
453	X			478			
454				479			
455				480	X		
456	X			481			
457	X			482	X	X	
458				484			
459	X			485	X	X	
460	X			486			
461				487			
462	X	X		488	X	X	

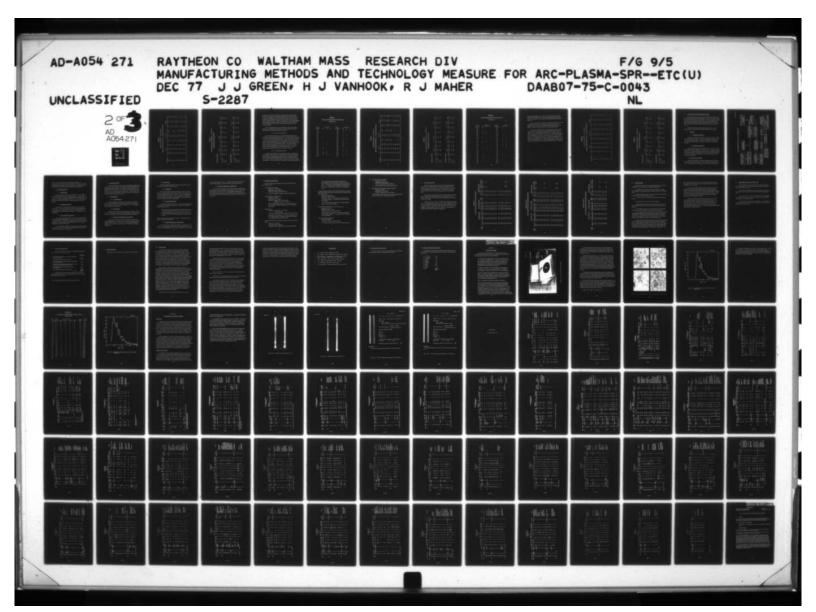




TABLE 17

APS PHASE SHIFTERS BATCH NO. 3

MICROWAVE MEASUREMENTS - ROOM TEMPERATURE

(•) NI *	5.45 5.7	-85 -85	-114 -109	-87 -88	-65 -60	-106 -102	+78 +90	- 3 + 4	+108 +120	+15 +21	REF REF
	5.2	98-	-1111	98-	99-	-106	+74	1 2	+104	+14	REF
3)	5.7	2.54	1	4.44	3.28		1.52	0.64	1.04	0.32	0.52
I. L. (dB)	5,45	1.98	> 6.5 -	3.84	2.94	> 7.0 -	0.40	0.72	0.58	0.56	0 64
	5.2	1.84	-	4.0	3.06	-	99.0	0.64	0.32	0.46	0 64
	5.7	392	382	374	391	374	360	391	339	383	20.4
Δφ (°)	5.45	393	384	375	392	375	362	392	340	384	200
	5.2	394	385	376	393	376	364	393	340	385	200
	Serial No.	442	444	447	451	452	462	476	482	485	480

APS PHASE SHIFTER ELEMENTS BATCH NO. 3 TABLE 18

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TEMPERATURE MEASUREMENTS

Serial No. APS 442

		5.2 GHz			5.45 GHz			5.7 GHz	
	-30。	25°	+85.	-30	25.	+82°	-30。	25°	+85
(dB)	6.04	1,84	2.44	2. 22	1.98	2.42	2.24	2.54	2.52
(°) ♦ △	519	394	325	503	393	326	492	392	329
(°) NI ф	313	REF	-130	229	REF	-117	179	REF	-108
Serial No. APS 476	APS 476								
		5.2 GHz		4,	5.45 GHz			5.7 GHz	
	-30。	25°	+85°	-30。	25°	+82°	-30	25°	+82.
I.L. (dB)	3.16	0.64	96.0	1. 10	0.72	1.16	1.0	0.64	1.72
(°) \$ \nabla	504	393	328	491	392	329	482	391	331
(°) NI ф	112	REF	-47	88	REF	-42	75	REF	-38

powder batch (LMTF 475 (G7)) onto the same substrate composition under very similar conditions. The weight of the finished units was very similar, 18.18 gm for APS 442 and 18.00 APS 476 indicating that the ferrite coating density must be essentially the same. The $4\pi \, \mathrm{M_r}$ values of 755 G and 756 G respectively further attest to a uniform ferrite density. This strongly suggests that the observed variation in insertion phase is due to causes not related to material properties but to air gaps at the waveguide interface or other instrumental causes.

Of the 22 sprayed samples lost in processing, 3 were too short after spraying, 4 were rejected for warping and 15 were rejected for excessive cracks in the ferrite coating again before machining.

2.4.4.4 Fourth production batch

In the fourth batch (Tables 19, 20, and 21) considerable difficulty was encountered with sample cracking much of which can be attributed to the use of a new batch of ferrite powder LMTF 475 (G8). In all, 145 samples were sprayed of which about half were rejected right after spraying and about one half of the remainder failed during machining or showed excessive cracks after the final anneal. Of the twenty samples chosen on August 9 for testing, 6 had $\rm B_r < 600\,G$ indicates they would have insufficient phase shift. Of the ten samples subjected to microwave testing on No. 564 had a phase shift of 322°. The insertion loss was very high on all but two of the test samples. Insertion phase variation was also very large, $\pm\,167\,^{\circ}$ relative to the mean.

2.4.4.5 Fifth production batch

In the fifth batch (Tables 22, 23, and 24) we were able to change from the G8 powder at about the half way point (APS711) to a new spray dried batch LMTF 475(G9). This powder gave considerably better results and produced most of the finished phase shifters APS711-765 which are listed in Table 22. To be specific, of the 122 samples sprayed, 43 were made with LMTF 475(G8) ferrite powder. Only one of these 43 (No. APS700), was processed through final

TABLE 19

APS PHASE SHIFTER ELEMENTS SELECTION

BATCH NO. 4

Units		Selection		Units	Se	election	S
Received	20	10	2	Received	20	10	2
404				604	· X ·	X	X
491			*	605			
492				609	X		
497				611			
498				615	X	X	
505	X			617			
506				618	X	X	
507				619			
508				622	X		
509				625	X		
510	X	X		627	X		
511				633	X		
512				634	X		
517	X			635			
541	X	X		636	X		
562				637	X	X	
564	X	X		638			
567	X	X		640	X	X	
593	X	X	X	643			
594				644	X		

TABLE 20

APS PHASE SHIFTERS - BATCH No. 4

MICROWAVE MEASUREMENTS - ROOM TEMPERATURE

	5.7	+ 176	09 -	- 146	REF	- 58	ω,	-107	107	+ 132	-170
φ in (°)	5.45	+ 165	- 62	-153	REF	09 -	6 -	-106	-106	+138	-167
	5.2	+ 162	- 62	-160	REF	09 -	- 10	- 105	-105	+ 144	-166
0	5.7	1.64		1.32	3.92	4.05	1.36	-	1	-	!
I.L. (dB)	5.45	0.88	8 dB	09.0	3.72	4.10	1.72	10 dB	8 dB	20 dB	20 dB
	2	90	-	0.52	3.88	4.20	1.80		-	-	-
	5.2	09.0	1	.0	e,	4	1.	1	-	i	1
•	5.7	362 0.	387	330 0.	360 3.	394 . 4.	400 1.	376	400	388	380
(ο) φ Q											
(∘) Φ∇	5.7	362	387	330	360	394	402 400	376	400	388	380

TABLE 21

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APS PHASE SHIFTER ELEMENTS - BATCH NO. 4

TEMPERATURE MEASUREMENTS

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1	0
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The same of the same of	No. APS 593
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	+82•	> 6.5	333	-47
5.7 GHz	25°	4.05	39.1	REF
	-30。	3.60	574	+93
	+85°	6.3	331	-47
5.45 GHz	25°	4.10	395	REF
S	-30。	3.56	562	+ 100
	+82.	4.20 > 6.5	329	-47
5.2 GHz	25°	4.20	393	REF
	-30。	3.60	546	+117
		I.L. (dB)	(∘) φ∇	φ in (°)

Serial No. APS 604	APS 604								
		5.2 GIIz		5.	5.45 GHz		t.	5.7 GHz	
	-30°	25°	+82.	-30°	25°	+82.	-30	25°	+82.
I.L. (dB)	> 6.0	1.80	1.40	3.54	1.72	0.86	2.72	1.36	.70
Δφ (°)	260	404	339	574	403	340	584	400	340
φ in (°)	1 58	REF	-136	- 143	REF	-115	+117	REF	-105

APS PHASE SHIFTER ELEMENTS SELECTION

BATCH NO. 5

20	Selection 10	ns <u>2</u>		Units Received	Se 20	lections 10	2
Χ.				734	X		
				7 36			
X	X			7 37			
X	X			7 38			
				7 39	X		
X				7 42			
				743	X	X	
X	X			744	X	X	
X				7 46			
				7 47	X	X	
X	X			750	X	X	
				754			
X	X	X		755			
				757			
				758	X		
X				760			
				761			
X				762	X		
X				764			
				765	X	Z	Z
	20 X X X X X X X X X X	20 10 X X X X X X X X X X X X X X X X X X	X X X X X X X X X X X X X X X	20 10 2 X X X X X X X X X X X X X X X X X X	20 10 2 Received X 734 736 X X X X 737 X X 738 739 X 742 743 X 744 X 746 747 X X 750 754 X 755 757 758 X 760 761 X 762 X 764	20 10 2 Received 20 X 734 X 736 X X X X 737 X X 738 739 X X 742 743 X X 744 X X 747 X X 750 X 754 X 755 757 758 X X 760 761 X 762 X X 764	20 10 2 Received 20 10 X 734 X 736 X X 737 X X 738 X 739 X X X 742 X X X X 744 X X X 746 X X X X 750 X X X X 755 757 758 X X 760 761 X 762 X X 764

machining and annealing. All of the other test samples were made with LMTF 475 (G9) ferrite powder. From the 38 final APS samples (APS 728 through 765 inclusive), 24 were brought to final dimensions and included in batch No. 5.

Clearly, the mechanical strength of the samples made with G-9 powder is superior to G-8 material. Physically there is less cracking in G-9 sprayed samples, and the ferrite coating density is higher and also more uniform, as reflected in sample weight of machined phase shifters.

The microwave measurements on 10 samples shown in Table 23 also indicate better reproducibility in phase shift insertion phase state and insertion loss. The average phase shift in batch No. 5 is 409.9°, which is about 30° higher than the average in batch No. 4. The standard deviation (S) in phase shift for the 10 batch No. 5 elements was 6.89°, which contrasts with S = 21.96° obtained for batch No. 4. Insertion loss measured on the batch No. 5 test samples is also decidedly improved over batch No. 4. Six out of the 10 samples have I.L. > 1 dB at center frequency. Insertion phase spread is less than half of the corresponding range in batch No. 4 samples.

TABLE 23

APS PHASE SHIFFERS BATCH No. 5

MICROWAVE MEASUREMENTS - ROOM TEMPERATURE

407 404 405 1.10 0.90 1.53 - 19 - 19 - 19 409 410 404 1.02 0.70 1.39 + 32 + 23 + 27 404 403 403 0.42 0.49 0.96 - 40 - 44 - 47 396 396 396 0.90 0.72 0.62 + 74 + 74 + 75 416 416 416 0.65 0.51 1.10 REF REF REF 415 414 413 2.50 2.80 - 66 - 66 - 66 413 414 410 5.42 5.38 5.60 - 50 - 50 - 50 417 417 1.01 0.90 1.37 + 9 + 5 + 3 414 414 410 1.40 1.18 1.82 - 32 - 31 - 30 414 414 410 1.40 1.50 1.82 - 32 - 31 - 31 414 416 416 1.60 1.50 1.8	Serial No.	5:2	Δφ (°) 5.45	5.7	I 5.2	I.L. (dB) 5.45	5.7	φ 2.5	φ _{in} (•) 5. 15	5.7
410 404 1.02 0.70 1.39 +32 +23 403 0.42 0.40 0.96 - 40 - 44 396 396 0.90 0.72 0.62 +74 +74 416 416 0.65 0.51 1.10 REF REF 414 413 2.50 2.30 2.80 - 66 - 65 414 410 5.42 5.38 5.60 - 50 - 50 417 1.01 0.90 1.37 + 9 + 5 414 410 1.40 1.18 1.82 - 32 - 31 416 416 1.60 1.50 - 25 - 23 - 23		407	404	405	1.10	0.90	1.53	- 19	- 19	- 19
403 0.42 0.40 0.96 - 40 - 44 396 396 0.90 0.72 0.62 + 74 + 74 416 416 0.65 0.51 1.10 REF REF 414 413 2.50 2.30 2.80 - 66 - 66 414 410 5.42 5.38 5.60 - 50 - 50 417 1.01 0.90 1.37 + 9 + 5 414 410 1.40 1.18 1.82 - 32 - 31 416 416 1.60 1.50 1.82 - 25 - 23		409	410	104	1.02	0.70	1.39	+ 32	+ 23	+ 27
396 396 0.90 0.72 0.62 +74 +74 416 416 0.65 0.51 1.10 REF REF 414 413 2.50 2.30 2.80 - 66 - 66 414 410 5.42 5.38 5.60 - 50 - 50 417 417 1.01 0.90 1.37 + 9 + 5 414 410 1.40 1.18 1.82 - 32 - 31 416 416 1.60 1.50 1.82 - 25 - 23		404	403	403	0.42	0.40	96.0	- 40	- 44	- 47
416 416 0.65 0.51 1.10 REF REF 414 413 2.50 2.30 2.80 - 66 - 65 414 410 5.42 5.38 5.60 - 50 - 50 417 417 1.01 0.90 1.37 + 9 + 5 414 410 1.40 1.18 1.82 - 32 - 31 416 416 1.60 1.50 1.82 - 25 - 23		396	396	396	0.90	0.72	0.62	+ 7.4	+ 7.4	+ 75
414 413 2.50 2.30 2.80 - 66 - 65 414 410 5.42 5.33 5.60 - 50 - 50 417 417 1.01 0.90 1.37 + 9 + 5 414 410 1.40 1.13 1.82 - 32 - 31 416 416 1.60 1.50 1.82 - 25 - 23		416	416	416	0.65	0.51	1.10	REF	REF	REF
414 410 5.42 5.38 5.60 -50 - 417 417 1.01 0.90 1.37 + 9 + 5 + 414 410 1.40 1.18 1.82 - 32 - 31 - 416 416 1.60 1.50 1.82 - 25 - 23 -		415	414	413	2.50	2.30	2.80	99 -	99 -	99 -
417 417 1.01 0.90 1.37 + 5 + + 5 + 5 + 5 + 5 + 5 + 5 + 5 + 5 + 5 + 5 + 5 + 5 - 31 - - 4 1 6 1 6 1 50 1 8 - 23 - - 23 - - - - 23 - - - - 23 - <td></td> <td>413</td> <td>414</td> <td>410</td> <td>5.42</td> <td>5.38</td> <td>5.60</td> <td>- 50</td> <td>- 50</td> <td>- 52</td>		413	414	410	5.42	5.38	5.60	- 50	- 50	- 52
414 410 1.40 1.13 1.82 - 32 - 31 - 416 416 1.60 1.50 1.82 - 25 - 23 -		417	417	417	1.01	0.90	1.37			+
416 416 1.60 1.50 1.82 - 25 - 23 -		414	414	410	1.40	1.18	1.82	- 32	- 31	
		416	416	416	1.60	1.50	1.32			

TABLE 24

APS PHASE SHIFTER ELEMENTS - BATCH No. 5

TEMPERATURE MEASUREMENTS

Serial No. APS 717

		5.2 GHz		5.	5.45 GHz			5.7 GHz	
	- 30.	25.	+82•	-30.	25.	+85.	-30	. 25	+85
I. L. (dB)	2.4	09.	.50	. 98	. 78	. 78	1.02	. 38	1.04
(∘) ∳∇	562	424	386	545	417	384	529	417	382
φ _{in} (°)	-40	REF	-128	-128	REF	-114	-178	REF	-100

Serial No. APS 765	PS 765							
		5.2 GHz		5.	5.45 GHz			5.7 GHz
	-30	-30° 25°	+82.	-30° 25°	25.	+82.	-30。	25.
I.L. (dB)	3.4	2.10 2.53 2.50	2.53	2.50	1.70	2.02	2.18	1.80
(•) ∳∇	541	420	374	526	420	372	512	412
φ _{in} (•)	-61	REF	-134	-144	REF	-124	-204	REF

2.40

374

-120

3.0 FLOW CHART OF MANUFACTURING PROCESS

The APS process for phase shifter manufacture has undergone numerous changes in the lifetime of this program but has evolved to a set procedure for the confirmatory and pilet production runs. The only changes between these two events concerned equipment modification to improve the alignment and sample clamping action with a graphite plug rather than the bare dielectric, and a rebuilding of the support structure to avoid sample wobble from these causes.

A general flow diagram of the process is given in Fig. 34. The diagram includes testing at the 50 percent level required by the contract.

3.1 Dielectrics

3.1.1 Production of dielectrics

Dielectrics are currently produced in the Research Division in 4 kgm lots by ball milling and calcining. Powder is isostatically pressed in 1 kgm bars and fired in electric kilns. Five kilns are available, each capable of firing two bars simultaneously. One man can process the 8 kgm needed in one month.

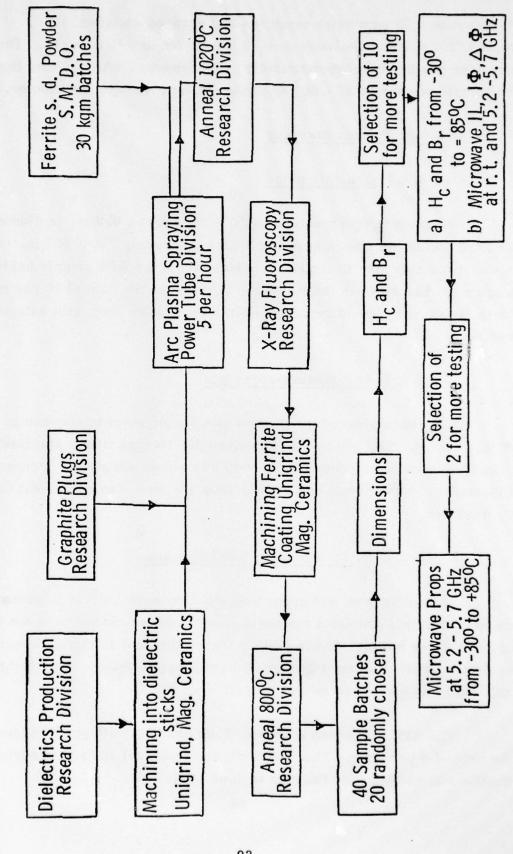
3.1.2 Machining of dielectrics

The fired bars are machined into sticks $0.060 \times 0.150 \times 7$ in. by one of two grinding shops: Unigrind, Inc., Dracut, Mass., or Magnetic Ceramics, Fairfield, N.J. Yield is typically 40 pieces per bar or 20 units for spraying. Three man-days effort are required per bar; ten are needed for 200 samples. The cost per dielectric pair is \$10.00. With a one-piece dielectric, this cost could be cut in half.

3.2 Ferrite Powder Production

The spray-dried powder is produced in 30 kgm batches by Raytheon Special Microwave Devices Operation. Seven batches were used on the contract. We typically spray 150 gm of powder to produce each sprayed boule.

FLOW DIAGRAM FOR APS PILOT PRODUCTION



At this rate a 30 kgm batch produces 200 sprayed samples, i.e., 30,000/150 = 200. Delivery on a 30 kgm batch is 6 to 8 weeks. The hours expended per batch is approximately 3 man-weeks, which places the cost of the ferrite powder at \$17.00 per phase shifter, a major cost factor.

3.3 Arc Plasma Spraying

3.3.1 Graphite plugs

We use graphite pieces about 0.8 in. long by 0.7 in. in diameter to hold the dielectric pieces at one end during spraying. A 0.007 in. taper is made of the diameter to facilitate release from the APS sample holder. The plugs were machined in the Research Division at the rate of 20 per man-day. Much faster rates could be achieved in a production shop with automatic tooling.

3.3.2 Arc-plasma-spray gun

The description of the plasma spray equipment is covered in Sec. 2 of this report. The gun is a Plasmadyne SG-1B type with a standard high-velocity attachment. The gun has a 40 kVa power supply and standard Plasmadyne gas controls and powder feed hopper. The equipment is owned by Raytheon.

3.3.3 Spray and upper holding ovens

The spray oven and upper holding oven were built at Raytheon using standard Kanthal flat plate elements, four 4×8 in. elements in the upper unit and three below. The hydraulic translation and dc motor driver rotation equipment were also put together by Raytheon personnel. The design and functioning is described in this report.

The APS equipment at Power Tube has routinely produced samples at the rate of 5 per hour. The COTR and the technical advisor observed a production run and verified the rate in June 1977.

3.4 Annealing at 1020°C

The sprayed coating must be heat-treated to bring the magnetic properties into line. This is done in electric kilns at the Research Division with programmed heating, soak time, and cooling to optimize properties. The standard anneal was a 100°C/m rise to 1015°C, a 4-hour soak, and cooling at 100°C/m, all in an atmosphere of flowing oxygen. With five kilns available, 200 boules could be annealed over one 3-day period. With production size kilns, that rate could be much greater.

3.5 Final Machining

3.5.1 X-ray fluoroscopy

Before committing the samples to final machining, we tested for flaws in coating or distortions in sample shape at the Research Division. Twenty samples per man-day was the production rate. X-ray testing may not be required in actual production.

3.5.2 Final grinding

Machining of coated samples to final dimensions of 0.220 \times 0.250 \times 5.145 in. \pm 0.001 is done at one or more grinding shops. Output per man-day per shop is 15 samples. Final machining cost was \$20.00 per element.

3.5.3 Removing machining stresses by annealing

The samples are annealed at 800°C at the Research Division to remove machining stresses. With five kilns available, 200 machined samples could be annealed over one 3-day period. The standardized schedule was 100°C/hour rise to 800°C, a two-hour soak, and a 100°C/hour cooling in stagnant air.

3.6 Sample Testing

Machined and annealed samples were produced in batches of 40, of which 20 were randomly selected for testing.

3.6.1 Dimensions

The external dimensions on the 20 samples were $0.220 \times 0.250 \times 5.145$ in. to within \pm 0.001 in. after the final anneal. Forty samples could be tested for dimension tolerance in 4 hours. We found that weighing the machined elements at this point was a very convenient way of monitoring the density of the ferrite coating. Density influences insertion phase of the device and should be monitored on all samples.

3.6.2 Hysteresis loop testing

At 15 amp-turns drive the coercive force and remanent magnetization were measured on the automatic loop tracer. Samples were required to meet the following criteria:

- a) Coercive force at room temperature will be such that 90 percent of differential phase shift is obtained at 15 amps drive current.
- b) Remanent magnetization at room temperature will be such as to produce at least 340° differential phase shift at 15 amps drive current.

Twenty samples could be measured per man-day. Ten samples from the 20 were selected for further testing.

3.6.3 Hysteresis properties vs. temperature

Coercive force and remanent magnetization were tested from -30° C to $+85^{\circ}$ C. B_r was well under \pm 10 percent over the temperature range, i.e., \pm 10 percent from the average value. Typical values for B_r were \pm 4 percent

at - 30°C and -8 percent at +85°C. A standard toroid was used in each run to verify reproducibility. Sample output was 10 samples per man-day.

3.6.4 Microwave properties vs. temperature

Two samples were selected from each batch for tests of phase shift, insertion loss, and insertion phase versus frequency (5.2, 5.4, and 5.7 GHz) and temperature (-30°C to +85°C). The temperature measurements are rather time-consuming and the output is therefore low: one sample per manday. This degree of microwave testing versus temperature would not be used in practice.

4.0 EQUIPMENT AND TOOLING

The following list gives pertinent data on each item used in the arcplasma-spray process.

1. Five Lindberg electric kilns

Design Cost: \$1,500 each

Replacement Cost: \$2,000 each

Ownership: Raytheon Research Division

Each oven is capable of firing two 1 kg bars simultaneously.

2. Grinding machines (outside vendor)

Design Cost: unknown

Replacement Cost: unknown

Ownership: Unigrind, Inc., Dracut, Mass.

Magnetic Ceramics, Fairfield, NJ.

Yield is typically 40 pieces per bar, or 20 units per spray. Three man-days! effort is required per bar; ten is needed for 200 samples.

3. Graphite plugs

Design Cost: \$2.00 each: \$.50 material,

\$1.50 machining

Ownership: Presently machined at Raytheon Research Division

Each plug holds one dielectric. The plugs are machined at the rate of 20 per man-day. Much faster rates could be achieved in a production shop with automatic tooling.

4. Ferrite powder production (ball mills, calciners, spray drier, tunnel kiln)

Design Cost: \$250,000

Replacement Cost: \$500,000

Ownership: Raytheon Special Microwave Devices Operation

The spray-dried powder is produced in 30 kg batches. We typically spray 150 g of powder to produce each sprayed boule. At this rate, a 30 kg batch produces 200 sprayed samples, i.e., 30,000/150 = 200. Delivery on a 30 kg batch is 6 to 8 weeks. Approximately 3 man-weeks are expended per batch.

5. Arc-plasma-spray equipment (Plasmadyne SG-1B gun with a Standard high-velocity attachment, spray oven plus controls, and upper holding oven plus controls)

Design Cost: \$20,000

Replacement Cost: \$25,000

Ownership: Raytheon. The spray and upper holding ovens were built at Raytheon. The equipment is located at Power Tube Division.

The arc-plasma-spraying equipment has routinely produced samples at the rate of 5 per hour.

6. X-ray fluoroscope (Radifluor 360, Torr X-Ray Corp.)

Design Cost: \$2,000

Replacement Cost: \$3,000

Ownership: Raytheon Research Division

Twenty samples per man-day were tested before being committed to final machining. X-ray testing may not be required in actual production.

7. Tools for determining dimensions:

Design Cost: \$100

Ownership: Raytheon Research Division

In 4 hours 40 samples can be tested for dimension tolerance.

8. Automatic hysteresis loop tracer

Design Cost: \$5,000

Replacement Cost: \$10,000

Ownership: Raytheon Research Division

Twenty samples can be measured per man-day.

9. Temperature measurements on magnetic properties

Design Cost: \$1,000

Replacement Cost: \$2,000

Ownership: Raytheon Research Division

The temperature measurements are rather time consuming and the output is therefore low: one sample per man-day.

5.0 DATA AND ANALYSIS

Hysteresis loop and microwave data on the 50 production samples that had the most thorough testing are summarized in Table 25. The coercive force at room temperature varies between 2 and 3.5 Oe, with no obvious dependence on ferrite density (col. 7). Phase shift also shows variation relative to $B_{\rm r}$ and ferrite density probably due to instrumental error.

Insertion phase shows fluctuations that are larger than we had hoped for. We expect that these fluctuations are due to variations in ferrite density, separations in the dielectric halves during spraying, and cracking in the ferrite coating.

Column 8 shows the range in B_r brought about by changes in temperature. The total percentage change from -30° to +85°C is the order of 12 percent, much smaller than the range in phase shift with temperature shown in the last two columns. In addition to $4\pi M_r$, phase shift is, of course, also dependent on other properties, such as k' and $4\pi M_s$. These factors can influence the temperature dependence considerably.

DATA AND ANALYSIS OF APS PRODUCTION RUN TABLE 25

	at 5.45 GHz C) (+85°C)	337		335													337	333			
Temperature Data	$\Delta \phi$ at 8 (-30°C)	535		292													535	525			
Tempe	ΔB_{r} (%) 30° + 85°	-7.7	-7.0	-7.9	-9.7	-7.0	-11.2	-9.3	-9.2	-7.8	-8.3	-5.4	-7.4	-8.9	-8.4	-8.3	-8.7	-8.9	-5.6	-9.2	-9.8
	△ B -30°	1.6	-1.2	4.6	3.1	3.9	3.8	3.1	4.1	2.3	2.1	2.1	3.5	3.3	3.2	3.2	3.1	4.6	1.5	1.1	0.4
sis	Weight (g)	1	:	-	:	1	:	1	:	:	1	1	:		!	!	:	:	:	:	!
Microwave Analysis	φ in 25°C)	-128	86 -	-131	ref.	9 +	- 44	+113	-116	96 -	-118	+	+ 82	+ 49	+ 49	- 17	ref.	- 20	- 81	+ 22	ر د
crowav	Δ φ° I. L. (5. 45 GHz,	0.78	1.16	1, 78	0.67	1.06	0.35	0.76	0.76	0.93	0.64	1.70	0.90	0.75	0.50	0.71	09.0	1.10	7.0	1.0	1.5
Mi		418	409	410	397	390	408	407	408	403	421	402	347	393	395	396	411	408	398	380	387
Hysteresis Data	$ m H_c$ $ m B_r$ (15 amp-turn drive)	765	092	168	922	753	787	719	782	717	782	771	190	782	788	787	820	807	730	714	750
Hyster	H _c (15 amp-	3.21	3.07	3, 22	2,44	3, 33	2.42	3.07	2.82	3, 38	3, 31	3.04	2.28	2.13	2.05	2,45	2,45	2.41	3, 22	2.78	2.76
	APS No.		331	346	353	354	358	373	383	386	388	390	394	397	399	408	411	412	424	428	431
	Batch No.	1										2									

TABLE 25 (Cont'd.)

DATA AND ANALYSIS OF APS PRODUCTION RUN

g]	Δφ at 5.45 GHz	(+ 82°C)	326						329								331	340				
Temperature Data	Δφ at	(-30°C)	503						491								299	574				
Temp	3 _r (%)	+ 85.	-8.3	9.7-	-8.7	-9.0	-8.8	-5.3	-6.8	-6.4	-7.4	6 .6-	-8.3	-5.3	6.9-	-7.7	-5.1	-8.6	-4.9	-6.4	-4.0	-3.6
	ΔB_{r}	-30	4.9	1.9	3.0	4.3	5.5	2.5	4.2	3.2	3.6	2.8	5.9	3.8	3.7	4.5	3.6	2.7	1.0	2.0	1.7	0.8
ysis	Weight	(g)	18, 18	18.43	18,36	18,34	18,31	17.46	18.00	17.14	17.89	17.96	17, 51	18, 58	17.18	18,30	18,34	18,69	18,75	18.97	19.01	18,98
Microwave Analysis	φ	25°C)	- 85	-114	- 87	- 65	-106	+ 78	က	+108	+ 15	ref.	+165	- 62	-153	ref.	09 -	6 -	-106	-106	+138	-167
icrowa	-	(5. 45 GHz,	1.98	6.5	3.8	2.92	7.0	0.40	0.72	0.58	0.56	0.64	0.88	8.0	09.0	3, 72	4.10	1,72	10.0	8.0	20.0	20.0
M	•	(5, 45	393	381	373	396	376	363	393	345	384	388	360	389	332	362	395	402	378	400	391	382
esis Data	ď	15 amp-turn drive)	755	725	719	767	716	269	756	689	692	774	707	715	642	664	100	219	597	640	603	613
Hystere	Ħ	(15 amp-1	2.64	3, 10	2, 99	2.96	3.05	2.67	2.70	2.69	2.66	2.61	2.17	2, 91	2.44	2.77	2, 91	2.81	2.86	2,83	2.79	2.82
		APS No.		444	447	451	452	462	476	482	485	488	510	541	564	267	593	604	615	618	637	640
	Batch	No.	က										4									

TABLE 25 (Cont'd.)

DATA AND ANALYSIS OF APS PRODUCTION RUN

		Hyster	Hysteresis Data	Mi	crowa	Microwave Analysis	sis		Tempe	Temperature Data	αI
Batch No.	APS No.	H _c (15 amp-t	H _c B _r APS No. (15 amp-turn drive)	Δ φ° (5.45	$\Delta \phi^{\bullet}$ I. L. ϕ in (5.45 GHz, 25°C)	φ in 25°C)	Weight (g)	△ B ₁	ΔB _r (%) 30° + 85°	∆ ¢ at 5 (-30°C)	Δφ at 5.45 GHz (-30°C) (+85°C)
2	069	3.09	731	404	0.90	-19	18.51	5.3	-8.9		
	693	2.70	782	410	0.70	+28	18,35	4.5	-9.9		
	711	2.69	742	403	0.40	-44	18.56	8.5	-11.5		
	714	3.18	748	396	0.72	+74	18,35	3.6	-5.5		
	717	2, 62	804	416	0.51	ref.	18,36	9.8	-10.6	545	384
	743	3, 30	713	414	2.30	99-	18.62	3.7	-8.3		
	744	3.30	703	414	5, 38	-50	18.78	1.6	-4.9		
	747	3, 41	737	417	0.90	+ 5	18,59	3.9	-11.7		
	750	3.25	732	414	1.18	-31	18.58	2.3	-6.3		
	165	3.46	727	416	1.50 -23	-23	18.75	0.8	6.9-	526	372

6.0 SPECIFICATION

As a result of the first article and pilot production runs, we would recommend three basic changes in the manufacturing process:

- 1) A change from the two-piece dielectric substrates to a single piece contingent on developing techniques to apply or introduce the switching wires,
- 2) Better methods of quality control on the spray-dried ferrite powder to give more uniform deposition characteristics,
- 3) Develop methods of collecting and reusing the ferrite overspray powder.

A one-piece dielectric would solve several problems:

- 1) Machining time would be effectively cut in half and yield per bar would be significantly increased (about 30 percent) because of reduction in kerf loss.
- 2) The tendency for any bowing or distortion would be significantly reduced because of the doubling in cross section of solid material.
- 3) The possibility of partial separation of the dielectric halves during spraying and the changes in insertion phase which result therefrom would be eliminated. We have noted a typical dielectric separation of 0.003 to 0.008 in. in the central two-thirds of most APS samples. The variation in this separation certainly contributes to insertion phase spread.

A second area where improvement would produce important dividends is better quality control on the spray-dried ferrite particle size and size range. During spraying, the particles are heated from room temperature to about 1500°C in milliseconds through coupling to the very hot plasma gases. The penetration of the powder into the plasma stream and the rapidity of melting or partial melting depends very critically on particle mass. The

more uniform the particle size, the more efficient this process becomes; large particles do not melt enough to stick and small particles overheat and volatiles (Li, Zn, O) are lost.

We have found through weighing experiments that the density of the deposited ferrite varies between samples sprayed with different ferrite powders. The density variation causes changes in thermal expansion match and may produce excessive cracking as in the LMTF475(G8) powder. Almost 75 percent of the samples sprayed with this powder yielded excessive cracking, yet we could detect no property differences to explain the problem. Better characterization tools are needed.

A third area needing improvement is reduction of ferrite powder losses during spraying. Losses are presently about 90 percent, i.e., of the 150 g sprayed, only 15 g remain in the machined phase shifter. Improvements in spray nozzle shape may increase the deposit efficiency. Another possibility would be recovery and reuse of over-spray powder. Reuse would be dependent on the degree of compositional change (volatilization) that has occurred during spraying.

7.0 REQUIREMENT FOR PILOT PRODUCTION

The processing and spray drying of the ferrite powder require the use of a 50 kg ball mill, a conventional calcining oven (850°C) and a spray drier with a capacity of 5 kg/hr.

Dielectrics production requires the same equipment at about 40 percent thruput level and, in addition, a periodic kiln for firing the dielectric bars.

The processing of the ferrites and the dielectrics for the production run made use at 5 percent of capacity of a 10,000 sq. ft. plant employing four skilled and two unskilled workers.

The arc plasma facility was contained in 600 sq. ft., making use of two skilled persons about one-third time. Annealing was done in one of two experimental size electric kilns.

8.0 COST FOR THE PILOT RUN

The cost breakdown for the various process materials and steps in the production run is summarized below.

		Unit Cost
1.	Spray dried ferrite powder, \$50.00 per pound and 0.33 pound per boule	\$17.00
2.	Processing cost for dielectric per pair	4.00 *
3.	Dielectric machining costs at \$245 per bar and yield of 25 dielectric pairs	10.00
4.	Plasma spray cost assuming setup and downtime and yield reduce production to three per hour	5.00*
5.	Machining of annealed phase shifter assuming 80 percent yield	25.00
6.	Two anneals plus inspection for dimension	2.00*
	Total	\$63.00

The hysteresis loop and microwave testing, apart from the temperature dependence tests, would add about 100 percent to the cost of each phase shifter tested in this way.

^{*} Cost is approximate and does not include overhead charges.

9.0 PROGRAM REVIEW

This subject is covered separately as an addendum to the final report.

10.0 CONCLUSIONS

The APS process has proved an effective and viable technique in fabrication of dielectric-loaded ferrite phase shifters of the Li-Ti type. The magnetic properties of plasma-sprayed materials generally compare favorably with conventionally fired ceramics. For example, all but one of the 50 microwave tested samples gave a differential phase shift greater than the 340° specifications, the average being 393° with a standard deviation of 20°. Insertion loss was < 1 dB on 25 of the 50 samples subjected to microwave testing and < 2 dB for 35 of the 50. In those instances where insertion loss was > 2 dB where additional testing was done, the loss could be reduced by a longer anneal in which oxidation would reduce the residual ${\rm Fe}^{+2}$ present. On the negative side the repeatability of insertion phase and the magnitude of coercive force (${\rm H_C}$) are not as good as conventional.

Insertion phase variation was about 2.5 times the goal of \pm 16° standard deviation, being actually somewhat larger than production by conventional means. The sources of insertion phase variation are 1) A variable void space between the dielectric halves produced by separation during spraying. This source could be eliminated by using a single-piece dielectric. 2) Changes in insertion phase due to variations in ferrite coating density, particularly when changing from one ferrite batch to the next. The correlation between observed variation in ferrite density (deduced from weighing machined phase shifters) and the corresponding changes in insertion phase, are shown for production batch No. 3 in Fig. 35. Since the dielectric has a known density of 3.98 g/cc, one can calculate ferrite density directly from the weight and dimensional data. A phase shifter weighing 19.15 gm would have a ferrite with 100 percent density (4.35 kc) whereas a phase shifter weighing 17.4 gm has a calculated ferrite density of 87 percent. The weighing of finished phasers is a rapid and convenient check on ferrite density and therefore insertion phase variation and should be included on a material specification in any future production.

The coercive force on APS samples is typically $2 < H_c < 3.5 \, \text{Oe}$,

consistently higher than these same Li-Ti ferrite compositions when conventionally fired ($H_c \sim 1$ Oe). The larger H_c is primarily due to porosity in the plasma sprayed materials which is typically 5 to 10 percent. H_c can be reduced to ≈ 1 Oe with Zn substitution, but at the expense of some temperature stability of the magnetization. A larger H_c implies a larger switching energy for the phase shifter driver circuit.

Apart from insertion phase variation and the larger coercive force, the most serious drawback of the APS process is cost. The process is not competitive at \$63.00 per finished phaser with the present garnet K-38 device. On the other hand, the costs could be reduced substantially by changing to a one-piece dielectric and by finding lower cost suppliers of spray-dried ferrite powders. The intrinsic materials cost of Li-Ti-ferrite is ~\$3.00 per pound and large-scale manufacturers should eventually be able to approach this cost within a factor of two, a 10 × reduction from small-scale manufacture.

The reuse of some or all of the overspray powder should lower this major cost item even more.

Machining costs are high because of the ferrite removal after spraying and will always create some disadvantage for the APS process.

It is important to mention the indirect benefits that have been gained by working with a new fabrication process. A good deal has been learned about the feasibility of applying the APS process to other ceramic and metallurgical coating projects of importance to the military. At present, four of these materials projects within the Research Division are competing for the use of the APS equipment. One project is to coat X-ray target anodes with a tungsten-rhenium alloy onto a high-temperature substrate. Cost savings over present vapor deposition processing could be considerable. A second project is to fabricate refractory oxide IR transmitting domes by

arc plasma spraying rather than by hot pressing or fusion casting. A third program would make use of plasma spraying to deposit electrodes for a TEA laser device. A fourth candidate is a project studying the activation of catalyst materials through melting oxide in different gaseous atmospheres at extreme temperatures. Finally, high-frequency phase shifter devices, which, because of the small coating thicknesses, may be fabricated advantageously by the APS process. We will continue to actively pursue these new technologies.

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11.0 PUBLICATIONS AND REPORTS

There were no publications or reports during the period associated with the research, study, or development under contract.

12.0 IDENTIFICATION OF TECHNICIANS

The following are the names of the personnel that worked on the contract and the total manhours performed by each during the interval covered by this report.

45
1503
63
75
149
27
2782
595
73
3578.5
8890.5

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APPENDIX I

Particle Size Analysis

The Zeiss particle size analyzer is a semiautomatic device for measuring and recording particle size on photographic prints or negatives. The device shown in Fig. AI-1 operates as follows:

An iris diaphragm, illuminated from one side, is imaged by a lens on to the plane of a plexiglass plate. An enlargement of the micrograph (transparent paper) is put on this plate. By adjusting the iris diaphragm the diameter of the sharply defined circular light spot appearing on the enlargement can be changed and its area made equal to that of the individual particles.

The different diameters of the iris diaphragm are correlated, via a collector, with a number of telephone counters, each counter corresponding to a certain aperture interval of the iris diaphragm.

When the measuring mark is equalized with a particle in the photograph, the footswitch is depressed. Thus the correlated counter is actuated, and a puncher marks the counted particle on the photograph. The photograph is then shifted until the next unmarked particle is above the stationary measuring mark, etc.

About 15 minutes are required for analyzing 100 particles.

Since the eye participates in the measuring process, the diameter of the particles to be measured in the photograph should possibly not be less than 1 mm. The instrument is provided with two measuring ranges. The first permits measuring particles of 1.0-9.2 mm diameter, the second such of 1.2-27.7 mm. The enlargement of the photograph should be in accordance with these limiting values. The particle sizes are divided into 48 continuous categories. In addition to the indicidual counters, which can be set back to zero, the instrument is equipped with a counter which registers the total of all counted particles.



Figure AI-1 The Zeiss Particle Size Analyzer.

The counting registers are arranged on a scale of exponentially increasing width. The scale is termed "relative" by the manufacturer in that the width of each counting interval is proportional to its size. The scale distorts the true distribution somewhat but gives more detail on the small particle end.

Counting data for photographs 1, 2, 3, and 4 are given in Table AI-1 for 24 particle size categories, showing an average diameter and the size interval for each. The sum of all particles within each range in the four photographs in Fig. AI-2 is given in the next column. These values were used to generate the histogram of particle diameter shown in Fig. AI-3. The average diameter for the 600 particles is 5.5 microns. The distribution shows a pronounced skewness. In the counting process we measured all resolvable particle aggregates whether or not there was apparent attachment to other particles. The distribution therefore indicates particle diameters which may be smaller than the actual distribution of free-standing particles.

The skewness towards larger particles in powder G2 suggests that it may be advantageous to remove larger particles by screening or air classification to give a more homogeneous size distribution for arc plasma spraying. In any event we now have a fingerprint of the size range for this powder which can be compared with subsequent batches.

One further exercise was performed with the particle count data. The small particles may be large in number and yet represent only a small weight fraction (or volume fraction) of the powder aggregate. Since we know the particles are hollow and have a wall thickness of 2.5 microns and can estimate a density of 2.5 gm/cc for the walls, one can calculate an average particle weight for each size category using the formula

wt (gm) =
$$\frac{\pi}{6}$$
 2.5 (d³ - (d - 2.5)³) × 10⁻¹²

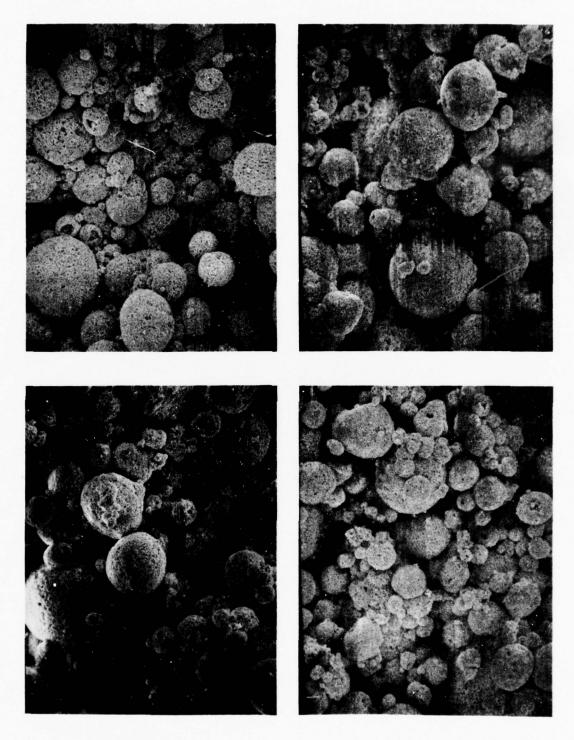


Figure AI-2 SEM Photographs at $400 \times$ of LMTF53(G-2) Spray Dried Powder.

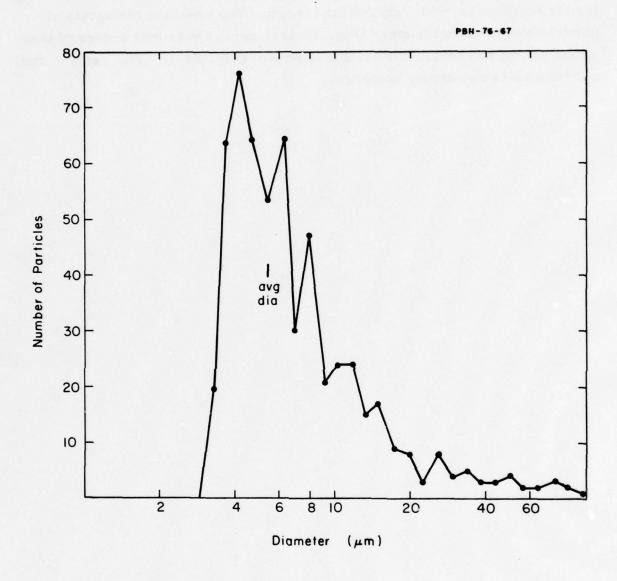


Figure AI-3 Histogram of Particle Size for Ferrite Powder LMTF53(G2).

The particle weights shown in Table AI-1 range between $46.37 \times 10^{-12} \, \mathrm{gm}$ for the smallest to $\sim 10^{-7} \, \mathrm{gm}$ for the largest. The resulting histogram of particle weight versus number (Fig. AI-4) is spread over four orders of magnitude versus two orders for the diameter plot (Fig. AI-3). The shape of the distribution is essentially unchanged.

PARTICLE SIZE DISTRIBUTION IN FERRITE POWDER
LMTF 53 (G2)

Avg.	Size		Particle C	ounts		Total	Avg. wt.
Size (Mi	Interval crons	Photo 1	Photo 2	Photo 3	Photo 4	Counts	× 10 ¹²
3.3	0.5	5	14	0	0	19	46.4
3.7	0.5	28	24	13	0	65	64.0
4.2	0.5	29	17	17	14	77	90.6
4.8	0.6	15	13	13	25	66	128.8
5.5	0.7	8	17	18	10	56	182.4
6.3	0.8	18	10	14	11	66	255.5
7.0	0.9	4	4	15	7	30	329.7
8.0	1.0	12	10	14	11	47	452.4
9.1	1.2	4	3	7	7 .	21	610.1
10.5	1.5	7	5	4	. 8	24	845
12.0	1.6	7	5	5	7	24	1140
13.6	1.6	5	3	7	0	15	1503
15.0	2.0	2	4	6	5	17	1861
17.6	2.5	3	0	3	3	9	2629
20	2.6	2	4	0	2	8	3457
22.7	3.0	1	1	1	0	3	4522
26	3.5	2	2	4	0	8	6019
29.5	3.9	1	2	0	1	4	7840
34	4.2	2	1	2	0	5	10535
38	5.0	1	2	0	0	3	13264
44	6.0	2	1	0	0	3	17950
50 -	6.5	0	2	1	1	4	23337
57	7.5	0	0	1	1	2	30519
65	9.0	. 0	1	0	1	2	39904
75	10.0	2	0	1	0	3	53403
85	12.5	0	2	0	0	2	68865
100		1				1	95741

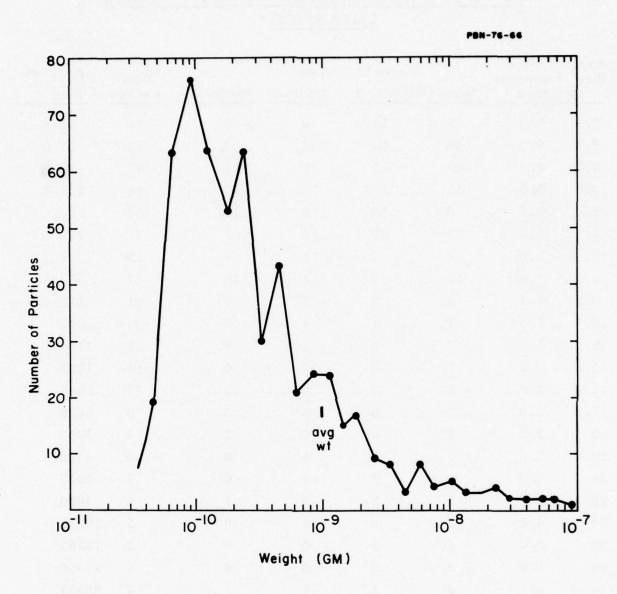


Figure AI-4 Histogram of Particle Weight for Ferrite Powder LMTF53(G2).

APPENDIX II

X-Radiography of Phase Shifter Elements

Introduction

The dissection of machined phase shifters from the confirmatory sample run had indicated a problem with warping, which resulted in uneven ferrite walls and low $B_{\mathbf{r}}$ and phase shift. With the exterior dimensions machined straight, any bowing or warping during spraying would produce walls thicker then the 0.50 in. dimension in some regions and a thinner wall on the opposite side. Very slight departures from straightness would have serious effects on $B_{\mathbf{r}}$. For example, a bow of 0.020 in. in the five-inch length would mean a thinning of one ferrite wall to 0.050 - 0.020 = 0.030 in. and, since the thin wall is flux limiting, $B_{\mathbf{r}}$ in this region would be reduced by 0.020/0.050, or 40 percent.

Although dissection of machined samples gives unequivocal evidence of warping, the procedure is destructive and is done after final machining, which itself is a costly step. There was, therefore, a strong incentive to develop a nondestructive process for evaluating wall uniformity in machined samples, and even greater incentive for finding wall thickness nonuniformities before the final machining. Some early experiments with X-ray and light transmission down the center slot showed promise but could not be made quantitative. Studies of X-ray fluoroscopy showed much better potential. We eventually adopted and used this technique for inspecting production run samples.

Experimental Technique

A conventional X-ray fluoroscope (Radifluor 360, Torr X-ray Corp.) was used to take the transmission photographs of the phase-shifter elements in two orthogonal directions. Samples were placed directly on Kodak-type M film and irradiated at 80 kV 3mA for 3 to 4 minutes with lead screen intensification. This produced full-size negatives with shades of gray, depending on transmitted intensity. Photographs of APS 251 and 258 are prints

of these negatives taken on two as-sprayed boules. The lighter areas indicate greater X-ray transmission.

The orthogonal views are shown in Figs. AII-1 and AII-2. H indicates that the join between the two dielectric halves is horizontal, and V indicates the verticality of this surface (perpendicular to the plane of the paper). In the latter, the join shows up as a thin white line where X-ray transmission is less impeded. The dielectric core with its machined center slot is also readily seen in these photographs.

X-ray transmission photographs have also been taken of machined elements as part of the analysis of phase shifters with low $B_{\mathbf{r}}$. In APS 143 (Fig. AII-3) we see that the dielectric is straight but $B_{\mathbf{r}}$ is nevertheless quite low. The reason for the low $B_{\mathbf{r}}$ in this case is machining error shown in the right-hand view, where one wall averages 0.039 in. rather than the 0.050 in. required. Assuming a $B_{\mathbf{r}}$ = 800G for a perfectly machined sample, we see in this case that machining error accounts for all of the observed reduction in $B_{\mathbf{r}}$. In Fig. AII-4 the X-ray shows a thin wall (left-hand view), this time brought about by a separation of the dielectric along the length which increased the core cross-section, reducing one ferrite wall.

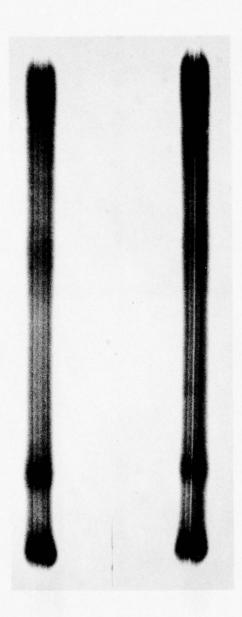


Figure AII-1 Orthogonal Views of Sample No. 257.

 \underline{H} \underline{V}

PBN-77-62

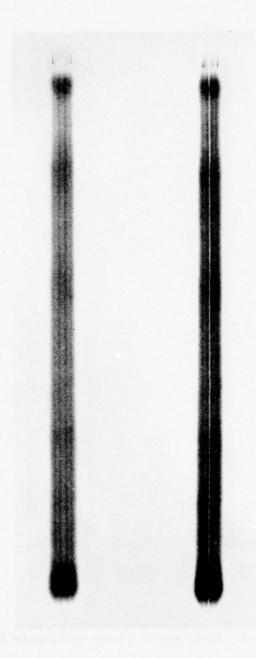


Figure AII-2 Orthogonal Views of Sample No. 258.

A. P. S. 143

 $H_c = 2.74$ $B_r = 626$ at 30 ampere turns.

Anneal: 1010° 1.5 hrs. O2; 800° 2 hrs. Air

Distortions:

Bow: .004 in.

Separation of insert halves: .006 in. for 2/3 length

Parallel to join 🔀

Wide-slot dimension:

Perpendicular to join

Thin-wall dimensions (mils):

End: 37

Center: 36

End: 45

Minimum: 36

Average: 39

Average thin-wall dimension as percentage of

ideal.050": 79 percent.

Estimated B_r based on cross-section: $B_r = 629$

COMMENTS:

Thin wall in strong direction due primarily to machining error.

Figure AII-3 X-Ray Transmission Photograph of APS Sample 143.

A. P. S. 146 $H_c = 2.73$ $B_r = 587$ at 30 ampere turns. Anneal: 1010° 1.5 hrs. O2; 1000° 1 hr. Air Distortions: Bow: .005 in. Separation of insert halves: .006 in. Parallel to join 🔯 Wide-slot dimension: Perpendicular to join Thin-wall dimensions (mils): End: 45 Center: 35 End: 40 Minimum: 35 Average: 40 Average thin-wall dimension as percentage of ideal . 050": percent. 80 Estimated B_r based on cross-section: $B_r = 640$

COMMENTS:

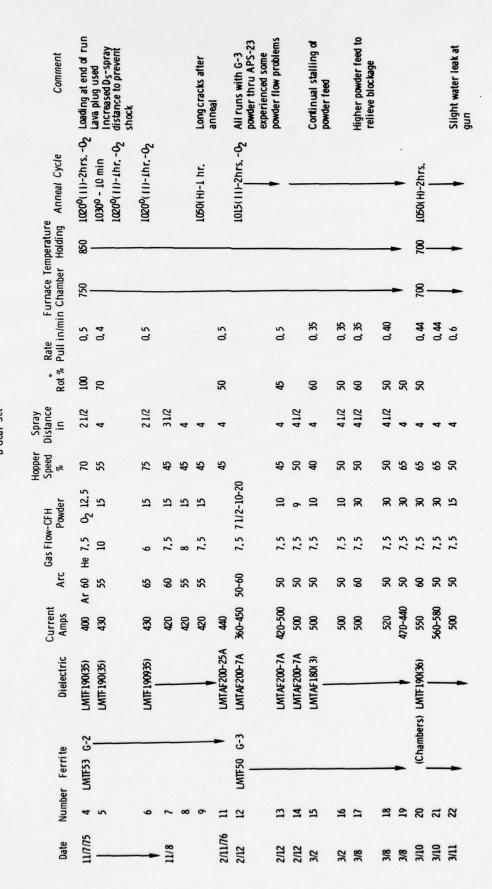
Separation along most of the length. Thin wall primarily machining error.

Figure AII-4 X-Ray Transmission Photograph of APS Sample 146.

APPENDIX III

Arc Plasma Spray Log

ARC PLASMA LOG High Velocity Nozzle B Gear Set

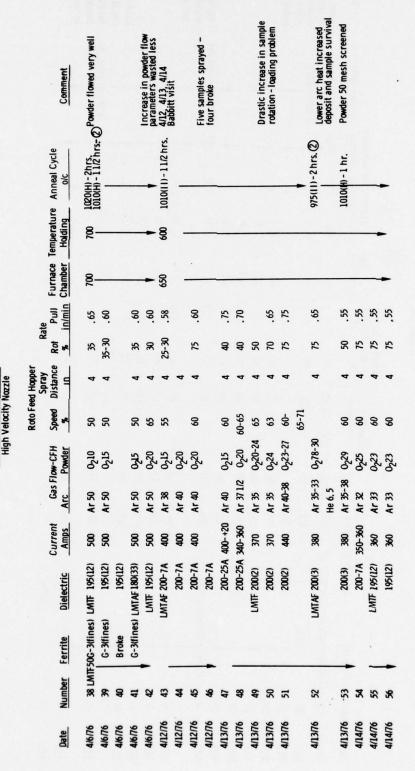


Tonas I

ARC PLASMA LOG (Cont' d.) High Velocity Nozzle B Gear Set

Comment	Powder dried over-	He gas elimination	might have contri-	מונפת וח את מונים				Approximately 160	grams of powder	sprayed per sample			Spitting still continu-	Ing at powder gas of 12 Too many samples breaking or fracturing	at base	
. Rate Furnace Temperature Rot % Pull in/min Chamber Holding Anneal Cycle	1050(H)-2hrs.	1050(H)-2hrs.			•	_	-	1050(11)-2hrs Air					_,	975 ⁰ (11)-2hrs. Air	-	
erature	90-		_		_	_	-	00.							-	
Furnace Temperature in Chamber Holding	00.					_	-	200	_						-	
te Furn Il in/min Ch	9.0	0.44		0.45	0,46	0.40	0,40	0, 40	·	0, 40	0,65	0, 48	0.65	0.65	0,65	
Rate % Pull i	20	8		40	40	20	40	40		32	32	35	8	8	8	
Spray Distance in R	4	4		4	4	4112	4	4112		4112	4	4	4	4	4	
Hopper Speed %	20	35		40	40	40	40	40		40	09	20	20	20	20	
-CFH Powder	02 20	12		12	12	10	10	10		01	01	10-13	15	15	15	
Gas Flow-CFH Powd	Ar 50 He 7.5 02 10															
Arc	Ar 50	20		22	20	29	20	22		20	20	53	55	55	20	
Current	450	410	Broke	410	410	530	470	480		440-500	200	500-550	575	520	520	
Diefectric	190(36)					LMTAF180(33)		180(33)								
	LMTF50(Chambers) LMTF190(36)	es)				LMTAF		LMTF50 G-3/Fines) LMTAF180(33)								Broke
Number Ferrite	MTF50(Ch.	(Fines)	_				-	MTF50 G-3							-	
Number	23 L	24	52	56	27	28	53	30 00		31	æ	33	×	35	*	31
Date	3/11/16	3/15	3/15	3/15	3/15	3/24	3/25	3/25		302	3/25	3/26	3/26	3/26	3/26	3/26

ARC PLASMA SPRAY LOG High Velocity Nozzle

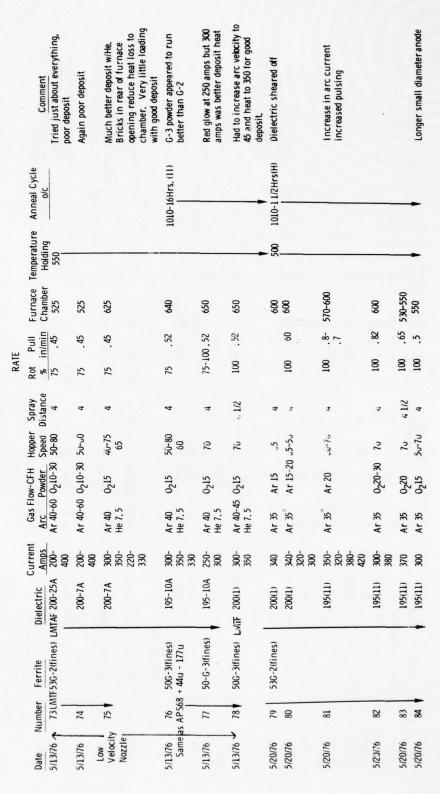


ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

	Comment								Deposit rate poor at 5 in.	appears to affect deposit more than simply increasing D _S						Change in powder did not change loading problem,	Changed anode from smaller bell shaped bottom to larger 901-11 which turned out to be a disaster for sprayability	Substrate rotation at slower speed, hotter substrate	Increased D _S required more arc heat
	Anneal Cycle 0/c	1010-1Hr(H)						-	1010-10Hrs. (11)				1010-10Hrs. (11)				Changed anode fr larger 901-11 whi sprayability		1010-16Hrs. (11)
	Furnace Temperature Chamber Holding	350						-	575			-	525						-
	Furnace	525-550	550	_				-	400				599-485	425-525		540	675		500-550
	ate Pull in/min	9.0	.75	.75	. 78	6.0	∞.	. 95	. 78		∞.	9.	99.	9.	. 95	1.0	9.0	75-40 .6	9.
2770	Rot Pr	75	75	22	75	85	83	82	15		80	80	75	75		75	75	75-4	75
יישיי ביוספול ויסדדור	Hopper Spray Speed Distance	4	4	4	4	4	4	4	5		5	9	4-5	9	4	4	4	4	2
	lopper Speed	09	09	09	09	09	99	09	09		09	09	9	22		2	09	02	8
	Gas Flow-CFH H Arc Powder	0,23	0,18	0218	0218	0218		8120	0218		8120	0218	0,18	0218-24		6218-28	0218-25	975	5 025
	Gas Flo Arc	Ar 33	Ar 33	Ar 33	Ar 33	Ar 33	Ar 33-35	Ar 33	Ar 33		Ar 45	Ar 45	Ar 33	Ar 33		Ar 33	Ar 40	Ar 40	Ar 38-45 0 ₂ 25
	Current	360	360	360	360	360	360	360	360		360	3 60-	380	380-	2 3	340	300	390	% of 05
	Dielectric	LMTF 195(12)	LMTAF200-7A	200-7A	200-7A	200-7A	200-7A	200-7A	LMTF 195(12)		195(12)	195(12)	195(12)	195(12)	ed 195(12)	195(12)	LMTAF 180(33)	50-G-3(fines) LMTF 195(12)	LMTF 195(12)
	Number Ferrite	57 LMTF50G-3(fines)												50G-3(fines) +325 - 80	Mesh screened	536-2	50G-3(fines) LMTAF 180(33) 50 mesh	50-G-3(fines)	
	Number	57 LM	58	65	09	19	62	63	B		9	99	19	89		69	02	11	22
	Date	4/14/76	4/14/76	4/14/76	4/14/76	4/14/76	4/14/76	4/14/76	5/4/76		5/4/76	5/4/76	5/4/76	5/4/76		5/4/76	911116	5/1/1/6	971716

F. . .

ARC PLASMA SPRAY LOG (Cont'd,) High Velocity Nozzle

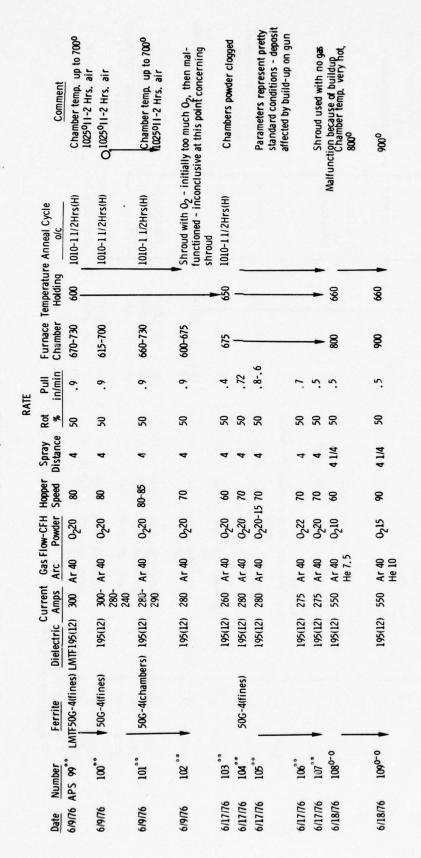


ARC PLASMA SPRAY LOG (Cont'd,) High Velocity Nozzle

	, and desired	Target area quite red at mini-	mum heat settings - still build- ing on gun end - minimum current settings 320 amps- 350 amps	No cracking due to wire insert	Sample broke after 11/2 in. of deposit			Target glow reduced to orange at higher velocity but powder flow had to be increased		New powder checkout chamber temp low			Chamber temp 575°- deposit much better	Motor fuse blew	Chamber temp up to 7000 010250 11-2 Hrs air)
	Furnace Temperature Anneal Cycle Chamber Holding	1-0101	1010-11/2Hrs(H)	1010-11/2Hrs(H)	1010-1 1/2Hrs(H)	1010-11/2Hrs(H)		1010-11/2Hrs(H)	1010-11/2Hrs(H)	1010-11/2Hrs(H)	1010-11/2Hrs(H)	1010-11/2Hrs(H)	1010-1 1/2Hrs(H)	600 1010-11/2Hrs(H)	1010-1 1/2Hrs(H)	
		1	69.					89.	86.	2 480-550		3 500-550	009		4 600-730	
RATE	Rot Pull % in/min	6. 001	-9. 001	00-100	6. 09	1.00-		-9' 001	-6. 001	100 32	9. 001	100 .63	100 1.1	50 .74	50 .74	
	Spray	4	4	4	4	4	inning	4	4	4	4	4 1/4	4	4	4	
	Hopper	20	50-70	02	02	70	ff in beg	02	8	02	02	70	92	55	20	
	Current Gas Flow-CFH Amps Arc Powder	0	0220	0220	0220	0220	Substrate sheared off in beginning of run	0220-23	0225	0225	0225	0,25	0225	0215	0215-20	
	Gas FI	Ar 40°	Ar 40- 50-60	Ar 50°	Ar 50 [*]	Ar 50°	Substra	Ar 55- 60	Ar 60°	Ar 40	Ar 40	Ar 40	Ar 40	220 Ar 40	Ar40	
	Amos			350		3%0		350	350 SE	320-	350	350	350		220-	
	Current Gas	LMTF50G-3(fines)LMTAF180(33) 340	Solid sub- strate w/one slot-external	Platinum wire forced into slot	LMTF 195(12) 360	Solid sub- strate with three slots	LMTAF 195-10A 360	195-10A 340- 350	LMTF 195(12) 320- 340	is) 195(12) 320- Ar 40 400	195(12) 350	195(12)	195(12)	195(12)	195(12)	
	Ferrite	WTF50G-3(fines								50G-4(fines)						ture of 43 psi
	Nimber	APS 85	·8	81,**	* 88	**68	06	91,**	76	93	8	8	8	16	86	Regulator pressure of 43 psi
	nate	9	6/1/76	9/1/19	9//1/9	6/1/76	9/1/9	9/1/19	6/1/76	911119	91119	91119	911119	9/16/9	91169	Re

Regulator pressure of 43 psi
Modified large opening anode w/Bell end

ARC PLASMA SPRAY LOG (Cont'd,) High Velocity Nozzle



**Modified large opending anode w/Bell end o-o Low temperature anode

ARC PLASMA SPRAY LOG High Velocity Nozzle

Comments	b. (H) New furnace - Works great - Chamber should be deeper - Anode worked poorly - Powder flowed poorly	1010 ⁰ -1 1/2 Hrs. (H) Chamber deepened -better				Changed from large anode to 901-12 anode (regular)		Big red glow - But best depositing conditions - even buildup negligible	(H) Powder flow a problem
Anneal Cycle	1010 ⁰ -1 1/2 Hrs. (H)	10100-1 1/2 Hrs						-	1010 ⁰ -1 1/2 Hr. (H)
Temperature Holding	0009	0009	009	009	009	009		009	.009
Furnace Chamber	8000	7250	750	750	750	750		750	750
Rate Rot % Pull in/min	∞.	.655	∞.	.7	∞.	. 92		8.	ш.
Rot %	92	22	20	20	20	20		20	20
Spray Distance in	4	4	4	4	4	4		4	4
	50-70	52-09	75	02	8	9		2	02
Gas Flow - CFH Hopper C Powder Speed	Ar 40 He 71/2 O ₂ 15 50-70	0 ₂ 15- 60-75 13	0 ₂ 13- 7 1/2	02 15	02 13	92 15	orted	95 15	02 15 70
Gas Flow Arc	Ar 40 He 7	Ar 40	Ar 40	Ar 50-45	Ar 40	Ar 35	Substrate cracked - aborted	Ar 35	Ar 40
Current	220-600		380	400-500	420	380	Substrate	400	400
Dielectric	MIF 195 (12)	LMF50G-4 LMF190(36) 320-340 fines							LMTF50G-4 LMTAF2007A 400 Fines
Ferrite	Chambers	LMIF 50G-4 fines							LMTF50G-4 Fines
Number	7115776 <u>A</u> 110	111 V S	∆ ∆ 112	△ △ 113	△ △ 114	115	116	117	118
Date	97/21/16	1116/76 △ △ 111	7	7	7				121

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing. △ 901-11 Anode modified to feed powder 1/4 in, cooler. △△ 901-11 Large opening cut back to 30º after powder port.

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

te Furnace Temperature Anneal Cycle Comments	1010 ⁰ -1 1/2Hr(H) Powd	0 600 1010 ⁰ -11/2 Hr(H) Powder freshly dried overnight @ 80 ⁰ C	0 600 Sample broke halfway	009	009 0	0 600 1010 ⁰ -1 1/2 Hr(H)	0 600 D _S decrease improved deposit	0 600 Holding furnace TC still not near samples	0 600 901-10 Anode did not work with usual parameters	0 700 1010 ⁰ -1 1/2Hrs(H)-0 ₂ Current too low- 180 amps	0 700 Temp, actually went to 1060 for 10 min.	700
Furna n/min Chamt	8 750	8 750	.83 750	.85 750	.85 750	.85 750	0 750	95 750	85 750	72 750	82 750	.87 750
Sa		.658					1.0	-40 0.95	0.85	0.72	-50 .7082	
ay nce Rot %	52	20	20	20	20	25	2 50	3 1/2 50-40	2 50	2 50	2 40-50	2 50
Hopper Spray Speed Distance	4	4	4	4	4	4	31/2	31/	3 112	3112	3112	3112
Hopper Speed	09	02-09	22	92	02	75	22	22	9	02	02	02
Gas Flow - CFH c Powder	ध १०	0 ₂ 15-	02 20	02 17	02 18	02 17	0517	02 17	6 17	02 17	02 17	0, 17
Gas	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40
Current Dielectric Amps	LMTAF180(33) 400	LMTF50G-4 LMTAF180(33) 400	400	98	300	LMTAF180(33) 300	300	LMTF50G-4 LMTAF180(33) 240 Fines	240-500	LMTF190(36) 200-220	220-240	230-240
		4-				1-4 LA		7-4 LV		1-4 LA		
Ferrite	_	LMTF50G			•	LMTF50G-4		LMTF50G Fines		LMTF50G-4 Fines		+
Number		120	121	221	123	124	125	126	127	128	129	130
Date	712176	702				7126/76				1/28		

901–10 Anode-powder port angled forward 55°.

Regulator pressure of 40 psi.

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

	Comments	Approx. 34 grams de- posited		Arc gas flow of 50 CFH - Too high	Approx. 121 grams G-4 powder per sample in this run	(A) Quick Anneal 135, 138 (D) 8000 - 40 min	② 1000º - 1 hour								``
	Anneal Cycle			-	1010 ^Q , 11/2 Hr(H) TC moved near	samples - Temp	1050 - 15 min 1010 - 1 hour				•	1010 ⁰ -1 1/2Hrs. TC located through	front brick		•
Furnace Temperature	Holding	700	200	002	200	002		002	200	200	750	200	200	0Ó.Z	700
Furnace	Chamber	750	750	750	750	720		750	750	740	740	750	750	750	750
œ	Pull in/min Chamber Holding	. 92	.95	.95	8.	.95		86.	1.0	1.0	6.	57.	.75	8.	ક.
	Rot &	20	20	20	20	20		20	20	20	20	20	20	20	20
Hopper Spray Speed Distance	٤	3 1/2		31/2	3112	31/2		31/2	3112	3112	3112	3 1/2	3112	3112	31/2
Hopper Spray Speed Distano	80														
활중		2	2	2	2	20		02	70	70	5 70	09	9	99	9
CFH	Powder	02 17	02 17	02 17	02 17	02 17		02 17	02 17	02 17	02 17-15	02 17	02 17	02 17	02 17
Gas Flow - CFH	Arc	Ar 40	Ar 40	Ar 50-45	Ar 40	Ar 40°		Ar 40°	Ar 40	Ar 40°	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40
Current	Amps	250	250	250-840	240	240-260		260	260	255	260	260	260	260	260
	Dielectric Amps	LMTF190(36) 250 		-•	LMTF190(36) 240						•	LMTF200(1)			
					6-4							6-4			
	Ferrite	LMIF50G-4 Fines		-	LMTF50G-4 Fines						•	LMTF50G-4 Fines			•
	Number	131	132	133	134	135		136	137	138	139	140	141	145	143
	Date	7/28			8/3							8/4			

ARC PLASMA SPRAY LOC (Cont'd.) High Velocity Nazzle

	Comments				Holding Furnace TC located on furnace floor with bead bent	into furnace area -	Samples broke because	of thermal shock			Holding Furnace TC malfunctioned - Temp too high		First sample sprayed from top down	New chamber elements - New powder dist, wheel		Only bottom to top spray in run - Only sample to crack in anneal
	Anneai Cycle	1010 ⁰ -1 1/2Hrs.		_•	No Anneal						No Anneal					1015 ⁰ -1 1/2Hr Air
Furnace Temperature	Holding	002	002	700	200	059	059	059	059	059	875-900	875-900	875-900	059	059	059
urnace I	hamber	750	750	750	200	200	200	002	200	200	902	200	200	002	902	902
Rate	ill in/min	æ.	=	1.5	1.0	1.0	1.0	1.0	195	1.0	0.95	0, 95	8.	.92	. 92	. 928
	% to %	23	20	20	52	20	20	20	50-40	20	20	20	20	20	20	20
Hopper Spray Speed Distance	اء	31/2	3 1/4	31/4	31/4	31/4	31/4	31/4	31/4	31/4	3 1/4	31/4	31/4	31/4	3 1/4	3 1/4 ing.
Hoppe	*	99	99	09	9	09	09	09	09	99	99	09	09	9	65	60 anneaí
GFH.	Powder	02 17	02 17	02 17	02 17	02 17	02 17	02 18	02 18	02 18	02 18 1/2	02 18 1/2	02 18 1/2	02 18 1/2	02 17	O ₂ 17-15 furnaces for a
Gas Flow - CFH	Arc	Ar 40	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 37 1/2	Ar 36	Ar 36	158 290 Ar 36 $\rm O_2$ 17-15 60 3 $^{\circ}$ (H) refers to APS holding oven. 11 and 111 refer to separate Lindberg furnaces for annealing.
Current	Amps	260-280	280	280	780	270	270	280	280	280	780	280	280	1/2) 290 5A(1/2)	A 290	290 refer to sep
	Dielectric Amps	LMTF200(1)	LMTF195(12)	→	LMTAF200(2)								-	LMTF190(36)(1/2) 29 LMTAF190-15A(1/2)	LMTAF200-7	II and III
	Ferfite	LMTF50G-4 I Fines		•	LMTF50G-4 LMTAF200(2) Fines								-	LMIF50G-4 LMIF190(36X1/2) 290 Fines LMTAF190-15A(1/2)	Dried 4 hrs. LMTAF200-7A 290 @ 100ºC	s holding oven.
	Number	4	145	146	147	148	149	150	151	152	153	154	155	156	157	158 fers to AP
	Date	8/4			8/26									8/31		(H)

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

	Comments			10 samples sprayed in	Z nrs. – approx. 1025 grams All 'downers''									Slight gun buildup at powder feed of 75		Top inch blown off but	Rotation erratic - slower	peads
ā	Anneal Cycle	1015 ⁰ -1 1/2Hr Air (11)	•	-		1015° 111-1 1/2Hr-02			1015 ⁰ (11)-1 1/2Hrs-0 ₂									-
Furnace Temperature	Holding	059	059	059	059	059	059	059	059	059	059	050	059	099	999	999	059	059
Furnace T	Chamber	200	200	200	002	002	200	200	200	989	200	700	200	700	200	200	700	200
Rate	Pull in/min Chamber	1.0	1.0	1.0	1.0	1.0	1.0	0.95	0.92	. 95	. 95 85	1.2	1.3	L3	1.3	1.3	1.3	1.3
	Rot %	20	20	20	20	20	20	50	50	20	20	20	20	20	20	20	45-50	20
Hopper Spray Speed Distance	Ë	3 1/4	3 1/4	31/4	3 1/4	3 1/4	3 1/4	31/4	31/4	31/4	31/4	65-70-31/4 80	31/4	3 1/4	3 1/4	31/4	31/4	31/4
Hoppe Speed	80	65	9	9	65	65	9	9	02-59	02	70	65-70 80	75	75-70	02	20	02	02
CFH	Powder	02 17 112	02 17 1/2	02 17 112	02 17 112	02 17 1/2	02 17 1/2	02 17 1/2	02 17 1/2	02 17 1/2	02 17 1/2	02 17 1/2	02 17 112	02 17 1/2	02 17 1/2	02 17 112	02 17 1/2	02 17 112 70
Gas Flow - CFH	Arc	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	175 + 305 Ar 36 O ₂ 17 1/2 70 3
Current	Amps	A 290	290	290	290	290	290	590	200	280	280-260	310	305	365	3) 305	305	A 305	302
	Dielectric Amps	LMTAF200-7A 290							LMTF200(2)	-•	LMTF200(1)				LMTAF180(33) 305	-•	LMTAF200-7A 305	→
	Ferrite	LMTF50G-4						-•	LMTF50G-4 LMTF200(2)	<u> </u>								•
	Number	159	160	191	162	163	164	165	166	191	168	169	170	171	172	173	174	175
	Date	8/31							1/6									

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

Comments	First sample after trying undried G-3 powder							Better deposit at 320 amps	Fastest deposit to date	No a smooth spray – first sample roughness	Very wobbly	First two substrates broke during spraying	Current crept up		stringers that are 1/2 in.	long then fall off Current surging during run	
Anneal Cycle	1015 ⁰ (111)-1 1/2Hrs-0 ₂							,	→	1025 ⁰ (11)-1 1/2Hrs-0 ₂ 			-	10150(111)-1 1/2Hrs-0 ₂		→	
Furnace Temperature Chamber Holding	059		050	029	999	999	999	999	999	059	059	059	920	059	999	059	
Furnace	200		200	710	710	200	002	700	700	200	200	700	700	902	902	720	
Rate Furnace Temperate Pull in/min Chamber Holding	7		1.0	1.0	1.3	1.3	1.3	1.3	1.4	1.0	.7-L0	13	1,3	1.0	7	8.	
Spray Distance in Rot %	3 1/4 50	31/4	3 1/4 50	31/4 50	31/4 50	3 1/4 50	31/4 50	3 1/4 50	31/4 50	31/4 50	31/4 50	31/4 50	31/4 50	31/4 50	31/4 50	3 1/4 50	f.
Hopper Speed	02 17 1/2 70	0 ₂ 17 1/2- 70 18 1/2	0 ₂ 18 1/2- 70 16	2	2	02	2	02	06	92	1/2 65	1/2 70	02 16 1/2 70	1/2 65	65	65 or anneali	
s Flow - CFH Powder	02 17	0 ₂ 17 81	0 ₂ 18 16	02 17	02 17	0917	11 60		0,17	02 16	02 16 1/2	02 16 1/2		02 17 1/2	02 17	O ₂ 18	and a second
Gas	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	Ar 36	200
Current Dielectric Amps	LMTF50G-4 LMTAF180(33) 300 Fines	LMTAF190-15A 300 	300	LMTAF180(33) 315	LMTAF190-15A 320	320	310	300-320	320	LMTF50G-3 LMTAF200(2) 320 Fines	LMTAF190-15A 330	LMTF195(11) 330-320	360	310	310	191 \Rightarrow 310-340 Ar 36 O ₂ 18 65 3 (H) refers to APS holding owen. If and III refer to separate Lindberg furnaces for annealing	
	F50G-4 U	_		_	_					ITF50G-3 L Fines	_	_				oven	
Ferrite	LMTFS								→	LMTES						holding	
Number	9/1	111	178	179	180	181	182	183	184	185	186	187	188	189	190	191 fers to APS	
Date	9/2/76									9/14						3	2. 411

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

Comments	Arc gas increased to	cneck surging - sample broke at base - Left in	chamber	Powder gas decrease stopped stuttering feed	Excellent parameters			Low arc gas tank volume	Powder ran out - Overlapped	in middle	Current surged		Current crept up	Excellent deposit	Current above 460 appeared to melt powder		pau	Attempt to similate APS 12 with higher velocity
Anneal Cycle	10150-11/2 Hr - O ₂			•					•	1200 ⁰ -30 min-0 ₂						•	1200 ⁰ -30 min-0 ₂ Controller malfunctioned	
Furnace Temperature Chamber Holding	059	059		059	059	099	999	999	599	059	059	059	059	059	. 059		059	
urnace 1	200	200		200	200	002	200	200	200	700	200	200	002	001	700		200	
Rate Furnace Temperate Pull in/min Chamber Holding	1.0	1.0		0.85	1.0	1.0	1.2	0.95	1.0	1.2	1.2	1,2	1.1	1.3	1.3		1.3-1.6	0.7
Rot %	31/4 50	31/4 50		31/4 50	31/4 50	31/4 50	31/4 50	31/4 50	3 1/4 50	3 1/4 50	31/4 50	31/4 50	31/4 50	31/4 50	31/4 50		31/4 50	31/4 45
Hopper Spray Speed Distanc	9	9		65	65	65	02	65	65	9	65-75	75	65	9	65		65-75	9
v - CFH Powder	02 18	92 18		05 17-10	% व	02 13	62 13	02 13	0213	02 13	02 11	02 11 1/2	02 11	02 11	0511		02 11	02 17-13
Gas Flow - Arc	Ar 37	Ar 37	roke	Ar 37	Ar 37	Ar 37	Ar 37	Ar 37	Ar 38	Ar 37	Ar 37	Ar 37	Ar 37	Ar 37	Ar 37		Ar 37	Ar 50
Current	320	320	Sample broke	320	360	360	340	350	350	340	340-400	400	400-460	440-460	460-500-		440	480
Cu Dielectric	LMTF195(11) 320		-															→
	3 LMT			4 LMT ers	_					4 LMT								
Ferrite	LMTF50G-3		•	LMTF50G-4 LMTF195(12) Chambers	-170 Mesh (-88µ)	_			_	LMTF50G-4 LMTF195(12) Chambers	r√88-	_						•
Number	192	193	194	195	1%	197	198	199	200	201	202	203	204	205	506		202	508
Date	9/14			9/15						12/6								

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nazzle

Comments	Too much waste overspray ed at hopper 65		New TC in holding furnace - Completely sleeved - located	hole - APS 212 appeared	warped after spraying	Looks warped			
Anneal Cycle*	1200 ^o (H)-30 min-O ₂ Too much wa Controller malfunctioned at hopper 65	-•	1020 ⁹ (H)-2Hrs-0 ₂						•
Furnace Temperature Chamber Holding	059	650	059	059	059	059	059	059	059
Furnace	700	700	200	700	700	700	700	200	700
Hopper Spray Speed Distance Rate Furnace Temperatu S <u>in Rot S</u> Pull in/min <u>Chamber</u> Holding	0.8	1.3	=	1.2	1.2	1.0	0.9	0.95	1.0
Rot %	\$	3	20	20	20	20	22	22	20
Hopper Spray Speed Distance	65-55 31/4 45	31/4	31/4	31/4	31/4	31/4	31/4	31/4	31/4
Speed	65-55	65	39	65	59	65	65	65	65
- CFH Powder	6,13	6,13	6,13	62 13	6,13	67 13	62 13	62 13	62 13
Gas Flow - CFH Arc Pow	Ar 50	Ar 38	Ar 38	Ar 38	Ar 38	Ar 38	Ar 45	Ar 45	Ar 45
urrent	480	044	A 360	A 420	400	400	400	400	400
Current Dielectric Amps	LMTF195(12) 480	→	LMTAF190-15A 360	LMTAF195-10A 420					•
Ferrite	LMTF50G-4 -88µ	-•	LMIF50G-4 -88µ	_					•
Number	500	210	211	212	213	214	215	216	717
Date	91/12/6		6216						

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

TABLE I
ARC PLASMA SPRAY LOG
High Velocity Nozzle

Anneal Cycle	1020 ⁰ (H)-2Hrs. 0 ₂ Anneal conditions	Air in hydraulic line	Current fluctuating as	Mothle forced slower	rotation rate Higher velocity affects sample rotation and eccentricity	1016 ⁰ (11)-3Hrs. 0 ₂ Substrate large grained - left in furnace during previous anneal	New anode and cathode for this day're run - flame firing upward - needed	more powder gas	1050 ⁰ -0 ₂ Quick anneal - no soak - broke apart - cathode check	1000 ⁰ (11)-5Hrs-O ₂ Sample fell in dismount	Seal leak at hopper			1000 ⁰ (11)-5Hrs-0 ₂ Powder buildup - broke two substrates		Left TC malfunctioning	1000 ⁰ (11)5 Hrs-0 ₂ Left TC failed		1000 ⁰ (11)5Hrs- ⁰ 2	Pull rate system will not hold set rate - continually	decreases		•	
ding						059	989	089		059	920	050		920	650	650	059	059	059	920	059	. 059	370	
	90 —				_	210	710	710	700	700	675	675		002	200		970	750	750	736	750	645	099	
Pull In/min	0.6	1.2	1.0	1.1	11	0.6	9.0	8-9.	0.8	0.6	1-0.6	1-0.6		9.0	0.6	0.6	0.7	0.8	0.8	0.8	0.8	0.9-1.0	1.0	
86	20	20	45	45	45	50	45	4	22	34	20	20		22	20	20	20	20	20	20	88	88	20	
=	3 1/4	3 1/4	3 1/4	3 1/4	3 1/4	3 1/4	3 1/4	31/4	31/4	31/2	31/2	31/2		31/4	31/4	3 1/4	3 1/4	3 1/4	31/4	3 1/4	31/4	3 1/4	3 1/4	
e e	59	65	65-75	65	9	02	65	2	99	99	65-70	02	_	9	50-40	20	83	69	9	9	65		65-75	
Powder	976	0214	0214	0,14	0,14	0213	0,25	0,25	0,25	05 30	0,17	0,17	Element Failed	0215	0,15	9,15	0220	0512	02 13-	0,22	0,22	0,23	0217-22	
Arc	Ar 38	Ar 38 .	Ar 38	Ar 45	Ar 45	Ar 38	Ar 38	Ar 38	Ar 38	Ar 40	Ar 40	Ar 40	Aborted -	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar38	Ar 38	Ar 38	Ar 38	Ar 38	
		00		20		9 5 5	0-340	-400	098	20	088	380	380	220	20	20	-400	920	096	330	20	965	020	
심	0-15A 3	4	8	4	4	0-15A	0(33) 45	×	0(33)	0-15A							0(2) 380	5-10A			0-15A	•••		
Dielect	MTAF19				-	LMTAF19	LMTAF18	-	LMTAF18	LMTAF19			-	LMTAF20		-	LMTAF20	LMTAF19		-	LMTAF19	-	LMTAF19	
il e	4756-5 1					4756-5	Ī		475G-5 -88	4756-5				4756-5			4756-5	4756-5					506-4	
	- MH				_	# —		-	LMI	W -			-	- WI		-	LMI	IM -				-	W -	
Number	218	219	220	Z	Ø	233	224	225	526	227	228	529	230	231	232	233	234	235	236	237	238	539	240	
~																								
The state of the s	Ferrite Dielectric Amps Arc Powder 1 In KOL Puil In/min Chamber Holding Anneal Cycle	LMTF475G-5 LMTF47	S8 ²	-88 [‡]	-88 ²	-88\$ -88\$ -88\$ -80 Ar 38 Qr 16 Sr 17 S	-88	-88\$\to 400	-88	Ferrite Dielectric Amps Arc Powder No. In No. Full Infinity Anneal Cycle -884	Ferrite Dielectric Amps Arc Fowder From Formal Chamber For	Ferrite Dielectric Amps Arc Fowder S 110 NOT S Full Infilmin Challing Anneal Cycle -884 400 Ar 38 O ₂ 14 65 314 50 0.6 700 650 1020 ⁰ (H)-2Hrs. O ₂ -884 400 Ar 38 O ₂ 14 65 314 45 1.0 -884 400 Ar 38 O ₂ 14 65 314 45 1.1 -884 400 Ar 38 O ₂ 13 70 314 42 6.6 710 650 1016 ⁰ (H)-3Hrs. O ₂ -884 380 Ar 40 O ₂ 25 65 31/2 42 0.6 700 650 1000 ⁰ (H)-5Hrs-O ₂ -884 380 Ar 40 O ₂ 30 65 31/2 42 0.6 700 650 1000 ⁰ (H)-5Hrs-O ₂ -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 650 -884 380 Ar 40 O ₂ 17 65-70 31/2 50 1-0.6 675 675 675 -884 380 Ar 40 O ₂ 17 65-70 31/2 675	Perrite Dielectric Amps Arc Powder No. In KOK Pruli Infinition Chalmer Front Profile Profi	MITATSG-5 LMTAF190-15A 300	Perrite Dielectric Amps Arc Fonder S 114 50 0.6 700 650 1020 ⁰ (H) 2Hrs. O ₂ 400 Ar 38 O ₂ 14 65 314 50 1.2 400 Ar 38 O ₂ 14 65 314 45 1.1 42 420 Ar 45 0.24 65 314 45 1.1 42 420 Ar 45 0.24 65 314 45 1.1 42 420 Ar 45 0.24 65 314 45 1.1 42 420 Ar 45 0.24 65 314 45 1.1	Perrite Dielectric Amps Arc Fower No. In ROLN Pull Infilling Challed Cycle -884	Minter Diefectric Amps Arc Fower No. In No. Fourier Four	Minter Diefectric Amps Arc Fower No. For For	Ministry Color Ministry Min	MINFAISC-5 LMIAFISO-15A 300	MIRATSG-5 UNITATION-LSA 300	MINTATSG-5 LMTAF190-L5A 200 Ar 38 Q ₂ 14 G ₂ 3 114 S ₂ 0 1.2 A A A A A A A A A	MINERISC-5 LMIAFIOL-15A 200 Ar 28 Q-15 65 3 114 50 L12 Ar 200-400 Ar 28 Q-14 65 3 114 65 L10 Ar 20 Ar 20 Q-14 65 3 114 65 L10 Ar 20 Ar 20 Q-14 65 3 114 65 L10 Ar 20 Ar 20 Q-14 65 3 114 65 L10 Ar 20 Ar 20 Q-14 65 3 114 65 L10 Ar 20 Ar 20 Q-14 G-15 Ar 20 Q-15 G-14 G-15 Ar 20 Ar 20 Q-15 G-14 G-15 Ar 20 Ar 20 Q-15 G-14 G-15 Ar 20 Q-15 G-16 Ar 20 Ar 20 Q-15 Ar 20	MINTERSOLD SA

AIII-15

ARC PLASMA SPRAY LOG (Cont'd,) High Velocity Nozzle

Comment		Sample dropped $\sim 1/2$ in.	during spray - overlap Ammeter of hopper @ 210 (420) milliamos		Jaws appeared to losen	Substrate wobbly -	G-5 Powder flowed better than G-4		Too much wobble for	First use of graphite Top keeper - unsuccessful	O ₂ Poor deposit		Broke in raising -	Tupper spray Broke - half sprayed - good deposit - hit obstruction		Powder flow improved as		O ₂ Spitting and too much wobble Blew gun out before spray	"Upper" spray - ran out of			Sample broke, upper spray	So much wobble that rotation	rate just moving - broke in chamber - left to cool	of substrate split	3,	
Anneal Cycle			No Anneal		•					-	1015 ⁰ (11)-2Hrs0 ₂			_	1015 ⁰ (11)-2Hrs0 ₂		•	1015 ⁰ (11)-2Hrs0 ₂	-	1015 ⁰ (11)-2Hrs0 ₂		-	No Anneal	-	Aborted because of substrate split	1015 ⁰ (11)-2Hrs0 ₂	•
Furnace Temperature Chamber Holding		450	485	995	059	059	672	059	675	350	059	059	059	059	920	059	009	009	009	720	720	710	710	725	009	009	009
Furnace		645	685		700	989	700	200	700	525	620	635	640	700	099	675	640	999	099	700	700	700	700	700	200	200	200
Rate Furnace Temperat Pull in/min Chamber Holding		0.8	0.8-0.9		1.0	1.0	1.0	6.0	0.7	1.0	0.4-0.7	0.565	0.5	0.8	0.8	0.8	0.8	1.0	9.0	0.5	1.0	0.9	0.8	0.8-0.6	0.8	1.0	
* 50		4	20	Sheared Off	8	20	9	4	8	8	22	20	20	20	20	20	20	20	20	20	22	22	20	50-30	20	45	20
Hopper Spray Speed Distance		31/4	31/4	Shea	31/4	31/4	31/4	31/4	31/4	31/4	3112	31/4	31/4	31/4	31/4	31/4	31/4	3 1/4	31/4	31/4	31/4	31/4	31/4	31/4	2 7/8	2 7/8	2718
Speed Speed		8 65-75	75		65	65	1 65	9	65-50	65	5 50-65	0 55	09 4	8	9	99	9	65	09	0 65-50	65	50-55	25	99	9	99	17-10 50-60
ow - CFH Powder	Broke	0,22-28	0,25	0,25	0,25	0215	0215-21	0,22	0218	05 30	0,15-25	0,25-30	0230-27	0530	0220	0220	0220	0216	91	15-30	23	22	22	22	. 13	13	11-11
Gas Flow - CFH Arc Powd	Dielectric Broke	Ar 38	Ar 38	Ar 38	Ar 38	Ar 38	Ar 38	Ar 38	Ar 38	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	Ar 40	9	9	9	40	8	9	35	35	125
Current		4 320	A 360	320	320	320	320	320	320	400	380	380	380	ed 420	350	350	350	350	350	360	360	360	360	300	220	240	230
Dielectric	F 200(2)	LMTAF 195-10A 320	LMTAF 195-10A 360							3-Solid	LMTAF 200(4)		_	Not Temp. Conditioned 420	AF200(4)	LMTAF195-10A	-	LMTF50G-4 LMTAF195-10A	LMTAF200(4)	AF200(4)				-	4F200(4)		
	G-5 LMT					3-5				-4 Fe ₂ C	LMT			t Temp.	-4 LMT			-4 LMT		3-5 LMT					3-5 LMT		
Ferrite	LMTF475G-5 LMTF 200(2)	-	LMTF50G-4		•	LMTF475G-5			-	LMTF50G-4 Fe ₂ 0 ₃ -Solid	-			N.	LMTF50G-4 LMTAF200(4)		-	LMTF50G	***	LMTF475G-5 LMTAF200(4) -88 #	_			-	LMTF475G-5 LMTAF200(4)	<u>8</u> _	-
Number	243	244	245	246	247	248	249	250	152	22	253	254	255	%	152	258	529	260	261	292	263	264	592	599	267	268	260
Pate	10/22		10/26							11/22					12/6			12/6		12/9					12/13		

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

Comment			Strate spill	Current too nign			Spray chamber a bit low	in temperature		Wobble ~ 1/8 in.	Wobble ~ 1/8 in. Broken	near base	Deposit thinned - "Upper"	spray	Substrate not preheated	Ran out of powder	Substrate separation on	initiating spray a continuous	problem	3/16 in. wobble initially but	improved with spraying - back	vent brick(Lge) replaced to con-	may have decreased - jerky	rotation	Blobs increasing -but deposit good-	no change made	No preheat for substrate - (NP)	Current fluctuated briefly	Current initially at 200 -	Short section for heavy deposit
Anneal Cycle*	1015 ⁰ (11)-2Hrs, -0 ₂		Apol ted because of surg			-	10150(11)-2Hrs-02									-	1015 ⁰ (11)-2Hrs-0 ₂	,	-	10150(11)-2Hrs-O ₂										•
Furnace Temperature Chamber Holding	009		009	009	009	002	059		009	009	009	009	009	002	009	009	009		009	009	009	009	009	009	009		009	009	009	009
Furnace Te	679		8	200	685	200	009		069	920	655	069	675	089	200	069	200		700	700	700	200	700	710	710		710	200	700	069
Rate Furnace Temperatu Pull in/min Chamber Hoding	1.0		1.3	1.3	1.1	1.1	1.0		1.0	1.0	1.0	1.0	0.8	1.0	1.3	1.2	1.0		1.0	7	1.0	1.2	1.1	1.1	1.0		1.1-1.5	1.2	1.1	0.7
ce Rot &	45		45	45	45	45	20		20	2 7/8 Fwd 50	20	2 7/8 Rev 45	20	45	45	45	20		20	20	45	45	8	9	40-35		35	35	35-30	8
Hopper Spray Speed Distance	2 7/8		2 1/8	2 7/8	2 7/8	2 7/8	2 7/8		0 27/8		2 7/8		2 7/8	2 7/8	2 7/8	2 7/8			27/8	2 7/8	2 7/8	0 27/8			5		2			-
	09	,	3	09	09	09	99		20-60	09	20	20	20	4	20	50	20		F50R	20	20	9-09	5	20	50-45		45-55	20	45	09
- CFH Powder	0,10		77	=	111/2	12	12		17	12	12	112	12-10	01	91	10	10		10	02 11	12	12	13	15	12	sit	12-15	15	15	15
Gas Flow - CFH Arc Pow	Ar 35		e e	35	35	35	35		35	35	35	35	35	35	35	35	35		35			35	35	35	8	Broke after 1/2 in. deposit	35	35	35	35
Current	200		300-270	250	220	210	240		210	210	210	210	210	210	210	210	210		210	210	210	220	220	220	220	Broke afte	220	220	220	220
Dielectric	VF200(4)					_	VF200(4)								Not Preheated 210	(NF)	F201-7A		_	LMTF475G-5 LMTAF201-7A							NP	NP	N N	NP
	_						3-5 LMT								Not	-	3-5 LMT/			3-5 LMT/	,									
Ferrite	LMTF 475(***	_	_	_	-	LMTF475G-5 LMTAF200(4)	1 88-				_				-	LMTF475G-5 LMTAF201-7A	₹88-	-	LMTF 4750	188-	_	_	_	_	_		_	_	-
Number	270		271	212	273	274	275		276	717	278	579	280	281	282	283	284		285	586		288	586	290	291	292	293	294	562	5%
Date .	12/13						12/16					A	III	[-1	7					12/21										

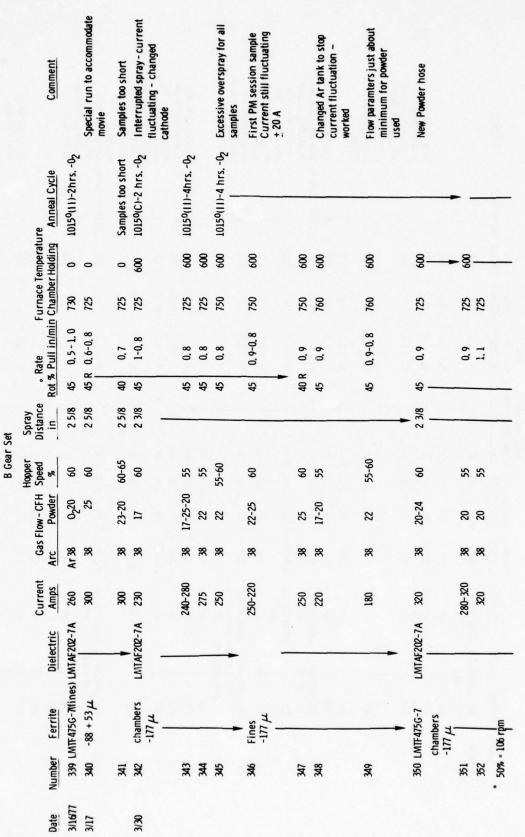
ARC PLASMA SPRAY LOG High Velocity Nozzle

		Comment	Chamber deepened - New Elements - 2 Pair 550W - 115ed	200	Rotation reversed at midpoint		Higher rotation to reduce substrate separation - ineffective		Retainer clip shift appears to reduce substrate separation			Heavy substrate Breakage with Powder Gas Initiation	"Upper" Spray		First Run with	Mounting Tube In- direct Drive Assembly and Suspension Plate- Tube jaws not tight	enough on substrate- Powder not dry enough New Tube Drive works well		First use of graphite substrate base and modified new mounting tube		Substrate separating upon initiating spray	
		Anneal Cycle	1015 ⁰ (11)-2Hrs-0 ₂						-			1015 ⁰ (11)-2Hrs-0 ₂		1015 ⁰ (11)-2Hrs-0 ₂					1015 ⁰ (11)-2Hrs-0 ₂		_	-
	Temo	Holding	009-006	059	959	920	059	920	059	059	059	059	650	920	009	009	009		009	009	009	009
	Furnace	Chamber	002	215	700	700	215	715	222	222	059	902	725	710	650	920	017		989	002	700	200
	Rate		6.0	1.0	1.0	1.0	1.0	1.0	=	0.95	0.0	6.0	40 1.1-0.9	0.95	0.85	0.85	1.0		1.0	1.0	1.0	0.85
e	8	86	83	33	F35R	35	09	₩	42	45	\$	45	9	8	22	8	æ	litions	\$	45	83	45
High Velocity Nozzle	Roto Feed Hopper	i	2 7/8							-	2 7/8			-	3 3/8			Shut down because of poor deposit conditions	25/8			-
High	Roto F	86	22	9-09	09	20	09-05	09-0	09	9	22	20	22	20	22	55	20	od po a	22	29	55-50	20
	Gas Flow-CFH		0215	15	13	15	15-20 5	15-20 50-60	50	20	15	15-17	15	15-12	52	50	20-25	wn becaus	50	20	20	20
	GACE	Arc	Ar35	33	35	35	85	35	85	33	33	8	37	35	33	88	88	Shut do	43	43	88	*
	Current	Amps	220	220-240	240	220	730	240	235	235	230	230	260-230	230	310-280	580	780	400	250	250	220	250
		Dielectric	LMTF 475 G-5 LMTAF 201-7A -88µ 60º Dry					-	•dN	N	-88µ 100° Dry NP	ď	N	NP	MTAF 200(4)			-	MTAF 200(4) NP	N	NP NP	ď.
		Ferrite	MTF 475 G-5 -88µ 600 Dry			_	,			-	LMTF 475 G-5 -88µ 100° Dry			-	309 LMTF 475 G-5 LMTAF 200(4)			-	313 LMTF 475 G-5 LMT AF 200(4) -88µ NP			-
		Number	76Z	298	586	300	301	305	303	3 6	305	%	307	308	309 LI	310	麗	312	313 U	314	315	316
		Date	1/6/77								1/12/17				2/10/77				2/16/77			

ARC PLASMA SPRAY LOG (Cont'd.) High Velocity Nozzle

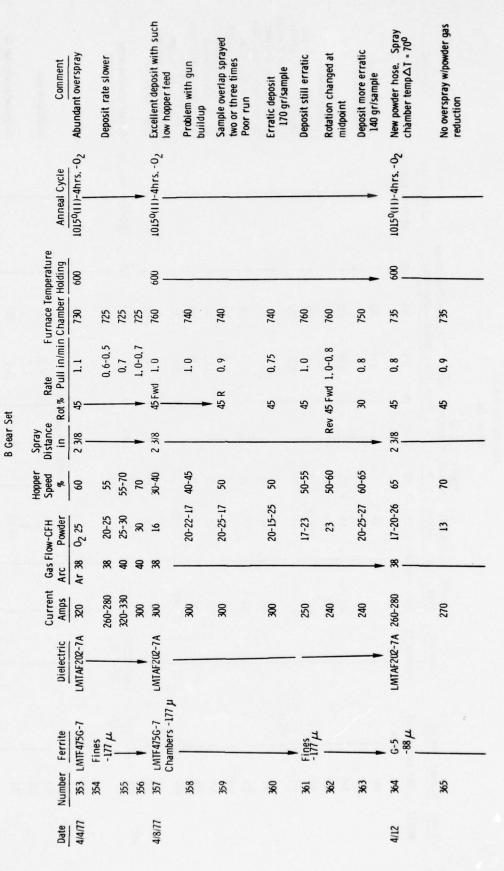
	, and an	Comment				Tube reject rod	Increased overspray exhaust with heat	reduction	Best run parameters for day		Substrate half cracked	-	size 10%-88u +53u, 60%-53µ +44, 30%-	44u. Powder flowing eratically. First use	of G-7 powder and uncoated elements in this series		220 amps appears to	be minimum for pow-	rent fluctuating at flow of 35 CFH of arc gas	Overspray reduced	O ₂ G-7 powder flow erratic at best, but hopper gas leak may	be the cause			Current crept up from 320 - 360	Conditions poor - hopper leak severe -	Graphite plug wobbly
	olon O leaded	Holding Anneal Cycle	-								-	1015 ⁰ (11)-2Hrs-0 ₂	_							-	10150(11)-2Hrs-0 ₂				_	-	
	Temp	HOIGING	009		009	009	009		009	009	009	009	009	009	009	009	009	009	009	009	009	009	009	009	019	009	
	Furnace	Chamber	200		720	521	725		725	009	600	780-740	092	022-092	725	760-725	725-755	735-760	730-740	745-765	82	730	730	730	730-745	730	
	Rot Pull	UI III	1.1		1.1	1.05	45 1.1-0.9		1.0	1.2	1.3	1.5	1.3	1.2	1.4	1.1	1.0	6.0	1.0	1.0	1.0	1.0	45 1.2-1.1	1.1	45 1.1-1.0	1.1-0.8	
e e	Rot	9	49		45	45	45		45	45	45	20	20	20	20	20	45	45	45	45	45	42	45	4	45	45	
High Velocity Nozzle	Roto Feed Hopper Speed Spray Distance	U.	-									2 5/8					-	3.0	2 5/8	2 5/8						-	
High		8	20		50-55	25	14 55-50-60		20	09-02	09	02	59-02	9-09	59	09	09	09	09	20	50-65	9	9	9	65	9	
		Powder	0215		15	15	14		13	15-10	13	15	20	20	16	15-20	20-25	20	20	20	17-30	8	8	8	8	8	
	Gas	Arc	Ar 38		88	88	88		88	88	88	35	35	35	8	88	88	35	88	8	88	88	88	88	88	88	
	Current	Amps	260		240	220	200		220	220	220	270	210-220	240	260	240	220	280	240-250	260-270	270-320	320	350	350	350	320	(NP)
		Dielectric	LMT	•dN	NP	LMTAF 201-7A NP	N Q		AN M	NP	NP	324 LMTF475G-7 LMTAF 200(4) -88 +53µ NP	LMTAF 200-7A NP	NP	327 LMTF475 G-7 LMTAF 202-7A -88 +53µ dried at 100º 4 hrs then screened NP	NP	NP	NP	NP	NP	333 LMTF475 G-7 LMTAF 202-7A -53 + 44µ NP	NP	NP	NP	d.	A N	No preheat (NP)
	:	Number Ferrite	317 LMTF475G-5	т/88-	_						-	LMTF475G-7 -88 +53µ		-	TF475 G-7 +53µ drie n screene	_			_	-	11F475 G-7		_	_		-	
		Number	317 LM		318	319	320		321	355	323	324 LM -86	332	326	327 LM -88 the	328	350	330	331	332	333 UV	334	335	336	337	338	
	;	Date	TIVZIZ									3/8			319						3/11						

ARC PLASMA LOG High Velocity Nozzle

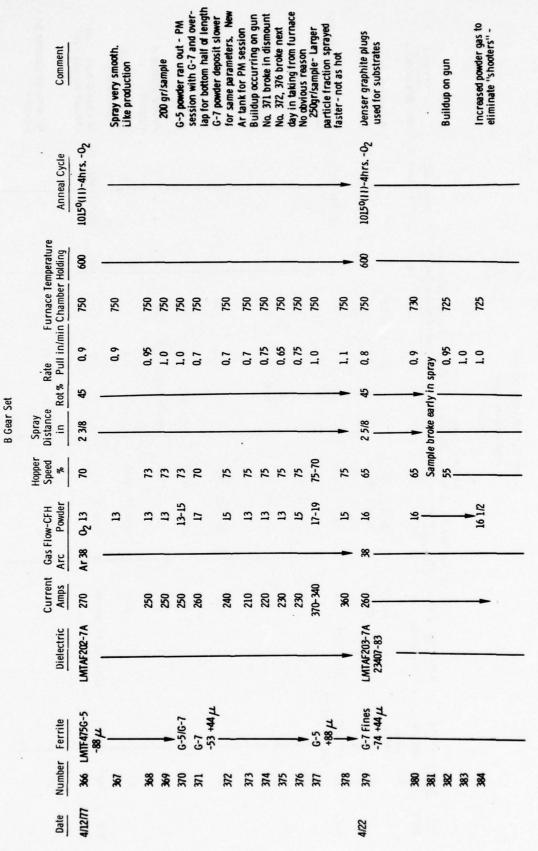


ARC PLASMA LOG (Cont'd.)
High Velocity Nozzle

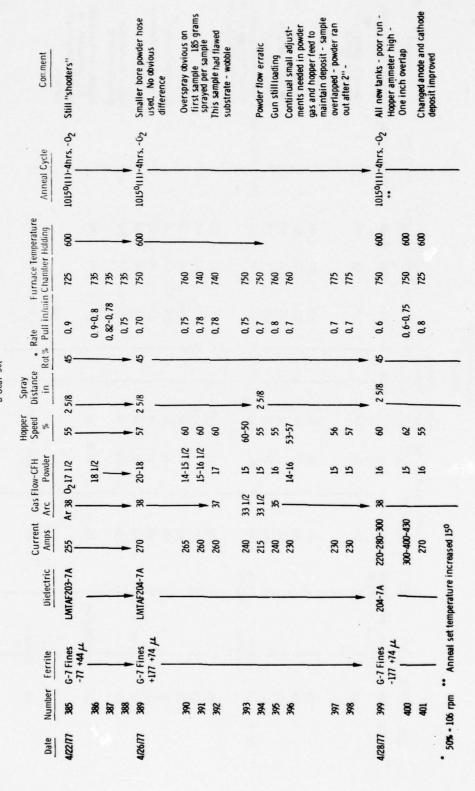
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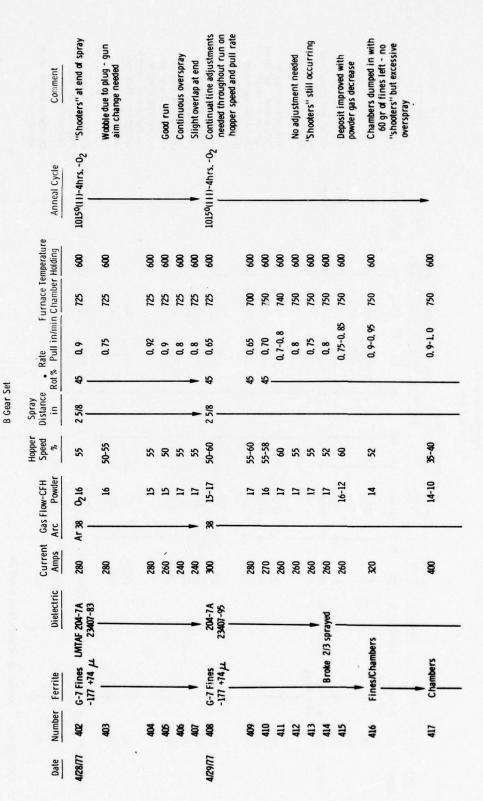
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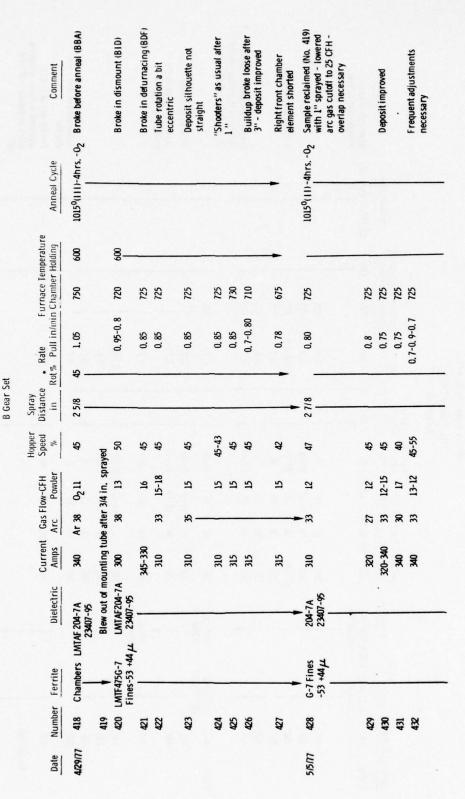
ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set



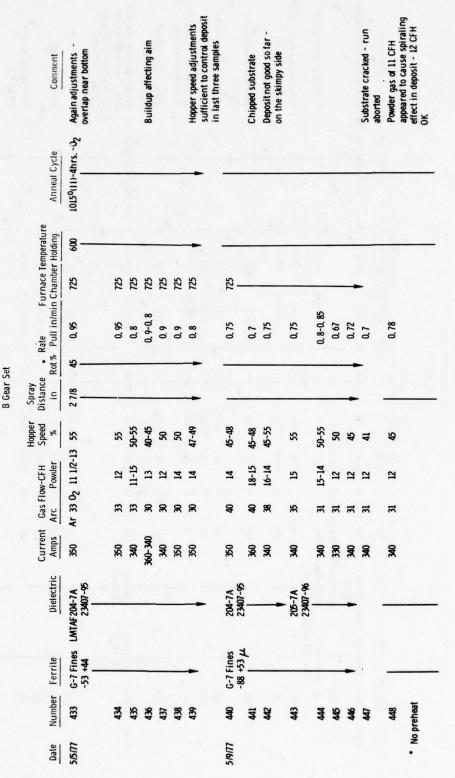
ARC PLASMA LOG (Cont'd.) High Velocity Nozzle



ARC PLASMA LOG (Cont'd.) High Velocity Nozzle



ARC PLASMA LOG (Cont'd.) High Velocity Nozzle



ARC PLASMA LOG (Cont'd.) High Velocity Nozzle

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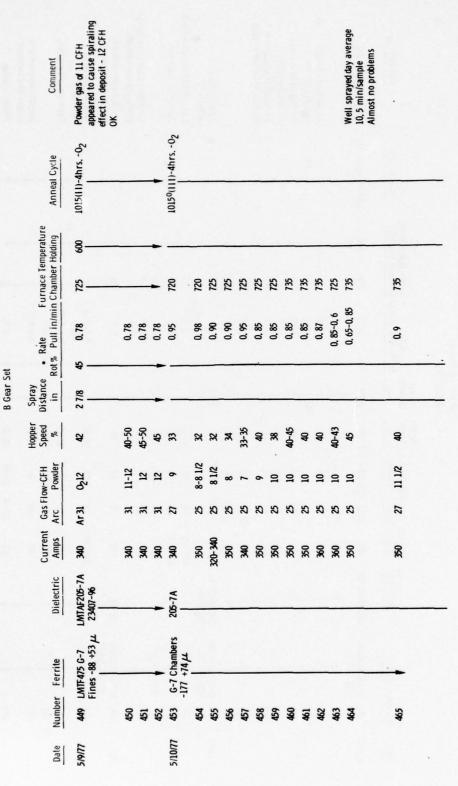
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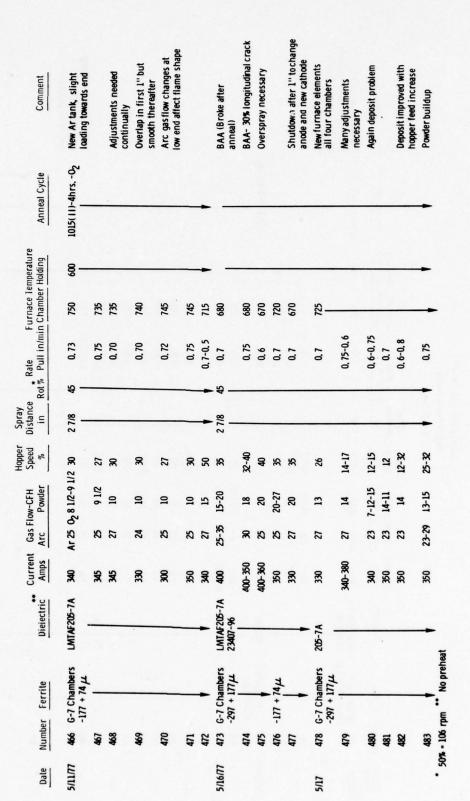
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ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set

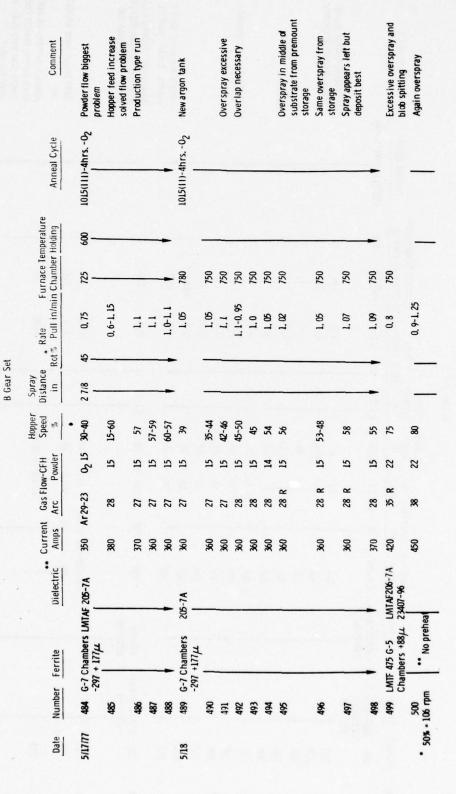


ARC PLASMA LOG (Cont'd.) High Velocity Nozzle

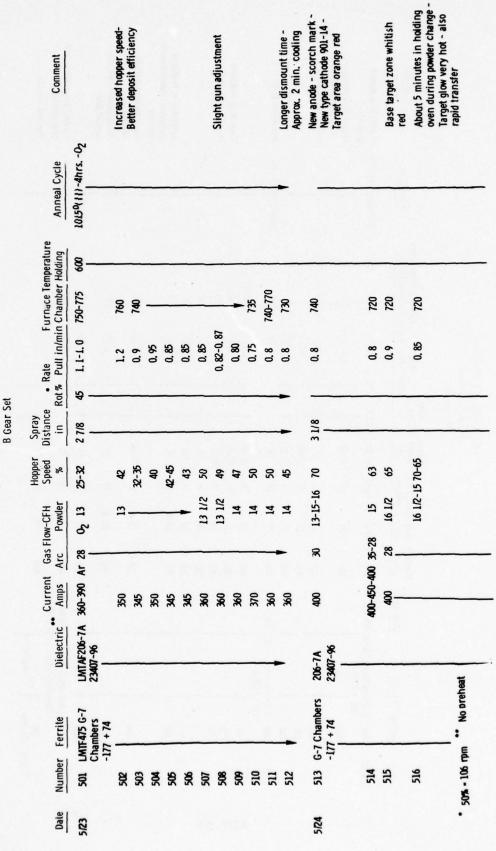
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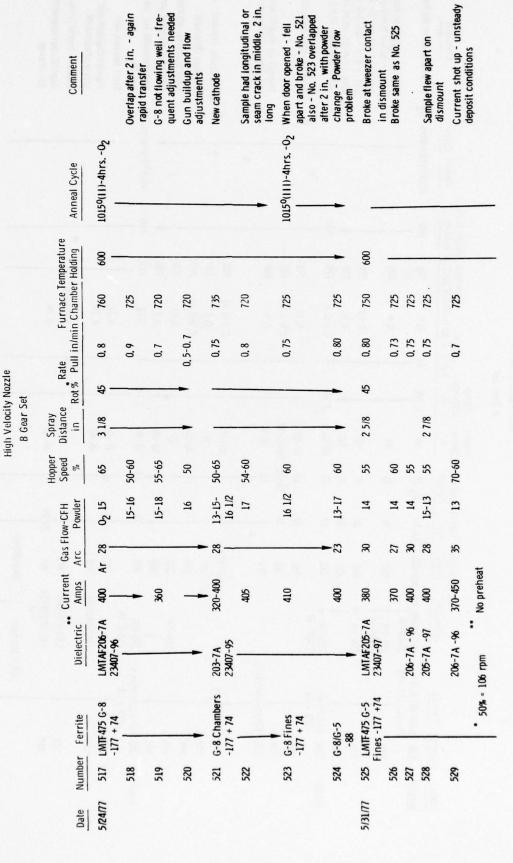
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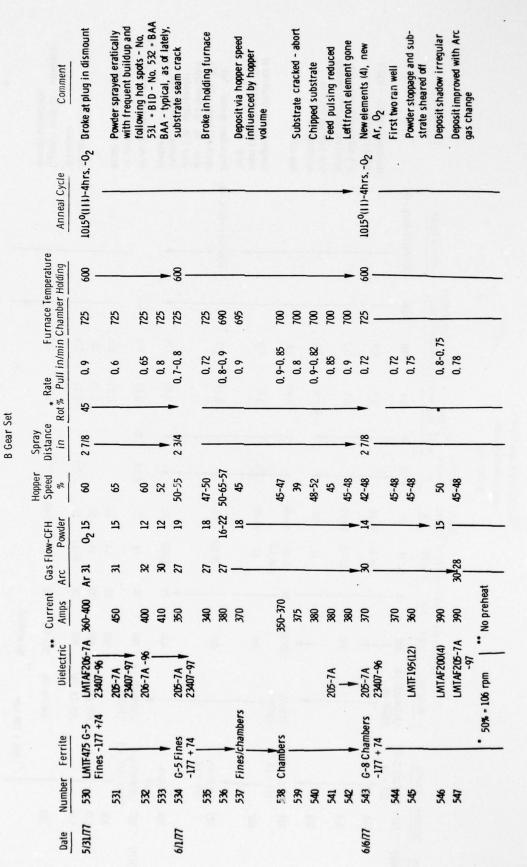
ARC PLASMA LOG (Cont'd.)
High Velocity Nozzle





ARC PLASMA LOG (Cont'd.)

ARC PLASMA LOG (Cont'd.) High Velocity Nozzle



Contract speed specification Silhouette much smoother out and restarted half-way demonstration (SSD) New cathode, anode Deposit stopped for clean-Ran out of G-8 after 1 1/2" Fast initiation of spray - adjustments throughout BAA -broke after anneal BAA - 80% longitudinal Broke before anneal Comment Last 2" very hot last two samples Quick dismount crack Abort 10150(111)-4hrs. -02 10150(11)-4hrs. -02 Anneal Cycle Furnace Temperature Rate Furnace Temperatur Rot % Pull in/min Chamber Holding 009 909 720 725 725 0.95-1.1 0.85 0.95 0.90 0.90 0,87 0.90 0.90 B Gear Set Distance Spray Ξ. 2 7/8 2 7/8 Hopper 48-45 50-53 45-50 47-50 % 47-51 47-53 50-48 51-55 50-45 45-47 48 47 8 53 Powder Gas Flow-CFH 0,15 5 5 5 5 7 15 Arc 8 8 8 8 Current Amps 380-320 370 390 390 370 380 380 370 LMTAF207-7A 370 23407-98 375 Dielectric LMTAF205-7A 23407-96 LMTAF205-7A 23407-97 205-7A -97 -177 + 74 μ 50% = 106 rpm ** No preheat G-5 chambers 552 LMTF475 G-8 Chambers -177 + 74 μ 548 LMIF 475 G-8 -177 + 74 µ Number Ferrite 562 G-5/G-8 563 G-5 chamt 550 551 555 553 554 556 558 559 261 557 260 Date 111119 11/9/9 8/9

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Plug broke off in dismount Aborted-dielectric cracked Excessive "shooters" - 40 arc gas better Broke in holding furnace mechanical performance End of SSD- excellent Aborted - per No. 580 Comment Abort after 1" crack 10150(111)-4hrs. -02 Test sample 10150(111)-4hrs. -02 Anneal Cycle e * Rate Furnace Temperature Rot % Pull in/min Chamber Holding 0.65 0.65 1.02 1.07 9.0 1.0 1.0 I.0 1.0 0.6 2 7/8 45=96rpm B Gear Set Spray Distance <u>u</u> 2 7/8 Hopper 45-52 16-16 1/2 47-50 50-55 50-52 % 99 8 2222 55 52 60 92 55 8 2 65 63 16 1/2 16-17 23 Arc Powder 16-17 20 02 16 Gas Flow-CFH 16 18 20 40-25 Ar 30 28 8 8 33 Current Amps 460-360 400-380 380 380 390 380 400 ** Dielectric LMTAF207-7A 23407-98 Fe₂0₃ Solid 207-7A 23407-98 205-7A 23407-97 No preheat Fines -177 +74 µ LMTF475 G-5 Chambers -177 + 74 μ 577 LMTF475 G-8 Number Ferrite 50% = 106 rpm 579 574 919 578 572 581 6/13/77 Date 11/8/9

well - breaking or cracking Substrate broke in middle Broke in holding brick set middle Broke per No. 586 All three samples sprayed Changed to SSD cathode arc improved - broke in Target hot -deposit slow Sample cracked before dismount - fell apart 10150(111)-4hrs. -32 Broke in defurnacing Stringers reduced at Reduced "shooters" Broke per No. 596 Comment Reason mystifying velocity increase Good profile Sporadic Anneal Cycle 8 Furnace Temperature e Rate Furnace Temperatur Rot % Pull in/min Chamber Holding 750 725 0.73-0.8 0.8-0.73 0.6 0.73 0.75 0.75 0.73 0.7 0.7 2 7/8 45=96 rpm 0.6 0.7 0.7 0.7 ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set Distance Spray Ξ 2 7/8 2 5/8 Hopper Speed 57-67 0/0 99-09 54-57 57-59 60-75 50-60 57-62 09 9 9 25 63 65 09 09 10-15 16-17 15-18 18-20 02 18 20 Gas Flow-CFH Powder 20 20 18 22 21 17 35-38 Arc Ar 35 * * * 8 8 35 8 8 8 8 8 Current Amps 300-280 340-350 360-320 285-295 380 290 380 360 360 380 360 300 310 Dielectric** LMTAF205-7A 23407-97 207-7A 23407-98 23407-98 205-7A 23407-97 205-7A 23407-97 205-7A 2347-97 No preheat Fines -177 +74 µ G-8 Fines -177 + 74 \mu 583 LMTF475 G-8 Number Ferrite -177 +74µ G-8 Fines : 50% = 106 rpm 286 595 230 585 588 589 592 593 594 265 28 591 28 6/20/77 6/14/77 6113/77 Date

Holding furnace door open to observe seam crack develop Abort Broke in furnace - fell over Sample left in spray cham-Flew apart - right element failed cracked Broke before door opened Broke in holding furnace Seam crack visible at dismount 38 velocity too high Comment Cracked at seam Still "shooters" 10150(111)-4hrs. -02 Shaky rotation New Ar tank 10150(111)-4hrs. -02 10150(11)-4hrs. -02 Anneal Cycle Survivors Rot% Pull in/min Chamber Holding 510 450 200 750 725 092 122 725 730 735 725 0, 78-0, 72 0.55 0.58 0.62 0.73 0.78 0.78 0.65 0.75 0.75 27/8 45=96rpm 0.78 0.7 0.7 Distance .⊑ Hopper Speed 55-60 29-65 55-60 57-66 57-63 60-64 60-58 89-62 65-67 8 62 62 8 30-38-28 16-18 19-21 18-20 02 17 Powder Gas Flow-CFH Arc Ar 38 Current Amps 310-325 310 8 LMTAF190-15A 310 305 350 贸 30 Dielectric LMTF475 G-8 Fines LMTAF205-7A LMTAF208-7A 23407-98 LMTAF190-15A LMTAF195-10A Fe₂0₃ - Solid LMTF200(1) 208-7A 23407-98 208-7A 23407-98 ** No preheat -177 + 714 H -177 + 74 pt Number Ferrite G-8 Fines G-7 Fines -44 50% - 106 rpm 809 28 909 109 209 603 80 99 909 910 119 612 613 209 609 6/20/77 6/22/77 6/27

ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set

Aborted - cracked substrate SS again noticed - only on last two New Ar O₂ tanks - Bottom clip Buildup caused erratic deposit - BID broke in Old style cathode 901-110 Substrate separation excessive (SS) Deposit very smooth SS in first 11/2" Seam crack at top Comment **Bottom** clip dismount 10150(11)-4hrs. -02 10150(11)-4hrs. -02 Anneal Cycle Furnace Temperature istance Rate Furnace Temperatu in Rot's Pull in/min Chamber Holding 770 725 0.72-0.77 0.73 0.75 0.72 2 7/8 45=96rpm 0.70 0.7 0.8 0.75 0.7 0.75 0.7 High Velocity Nozzle B Gear Set Distance Hopper Speed 58-68 02-09 9-09 65-70 9-09 60-65 49-60 60-65 9 9 9 15-20 20-22 23-25 Dielectric Amps Arc Powder 22-23 20-25 02 20 20 20 22 22 22 38-35 1/2 LMTAF195-10A 305-330 Ar28-38 .58 8 8 * 8 365-370 380-420 360 370 410 380 380 410 420 410 400 395 400 LMTAF200(1) 190-15A 195-10A 190-15A 208-7A 208-7A -100 195-10A 200(4) No preheat Fines -74 + 44 μ 614 LMTF475 G-8 -177 +74 JL Number Ferrite -177 +74 pt 617 G-8 Fines G-8 Fines 50% = 106 rpm 615 919 625 620 819 619 624 621 622 623 626 627 628 629 6/28/77 6127177 Date 6219

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ARC PLASMA LOG (Cont'd.)

Aborted - dielectric cracked No evidence of seam crack throughout day's run Looked warped at dismount Long crack upper half -taken hot from holding furnace Stringers causing target temperature to change -Skimpy substrate half Broke in defurnacing Temperature at target 1350^oC Comment 1_T = 1390°C 10150(111) -4hrs. -02 10150(111)-4hrs. -02 10150(11)-4hrs. -32 Anneal Cycle e * Rate Furnace Temperature Rot % Pull in/min Chamber Holding 89 735 745 740 260 0, 75-0, 72 0.74-0.78 0.78 0.72 0.82 0.83 0.88 0.75 0.8 0.82 0.75 0.8 8.0 0.8 0.7 High Velocity Nozzle B Gear Set Spray Distance __ Hopper Speed 60-63 60-65 60-64 19-09 9 Powder 02 20 24 Gas Flow-CFH Ar 37 Amps A 335-355 370 90 8 38 88 365 370 355 33 345 355 350 355 8 LMTAF190-10A 24650-1 Dielectric LMTAF195-10A LMTAF200(4) LMTF200(2) 195-10A 208-7A -100 No preheat I LMπ475 G-8 Fines -177 +74μ 6-8 Fines -177 + 74 μ -177 +74 µ G-8 Fines Number Ferrite 50% - 106 rpm **₹** 632 633 634 635 638 639 3 3 3 643 647 636 637 B 8/4/77 Date 679 06/9

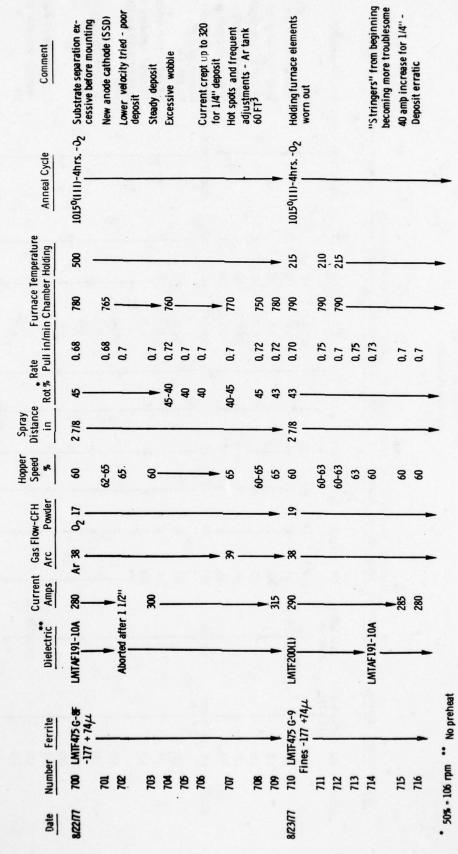
ARC PLASMA LOG (Cont'd.)

good sample - stuck in tubedismount - broke in de-furnacing Stringer length increasing Many adjustments plus two No. 658, 659 to try and eliminate substrate separa-Current fluctuation I" from bot. - deposit hotter Only 1/4" bite in plug for Many adjustments in last Shadow of seam crack at Smoother run but broke Shut down and cleaned Broke in dismount but two samples, 659, 660 Broke in defurnacing Powder running out Sub cracked - abort appeared very hot Comment in defurnacing than usual overlaps 10150(111)-4hrs. -02 Anneal Cycle e * Rate Furnace Temperature Rot % Pull in/min Chamber Holding 9 770 740 740 0.75-0.80 0.75-0.80 0.78-0.85 0.78-0.83 0.85-0.80 0.65-0.70 0.9-0.75 0.85-0.9 0.73 0.88 0.78 0.75 0.91 0.7 0.7 0.7 B Gear Set Spray Distance Ξ. 2 7/8 Hopper 65-70 65-70 67-72 9-09 60-65 60-65 60-63 9 63 65 2 9 9 9 65 02 24-26 Powder 23-20 20-21 2 2 Gas Flow-CFH 92 92 24 20 21 22 Ar 38 Arc 8 8 8 Current 380-400 390 2 2 330 420 \$ 6 LMTAF190-10A 24650-1 Dielectric** * 50% = 106 rpm 1 No preheat Fines -177 + 74 µ LMTF475 G-8 Number Ferrite ₹ 649 650 651 652 653 655 959 658 099 299 699 99 159 199 Date 8/4/77

Seam crack before dismount-Broke while spraying - seven in a row of new 191-10A Slight bow in substrate half Excessive separation - broke Broke in chamber 1" from Seam crack appeared after two samples sprayed More "shooters" this run Broke after 2" as No. 675 Broke at base - stored in No. 3 position Control check sample Comment **Broke in dismount** More adjustments Same as No. 671 Target very hot completion 10150(111)-4hrs. -02 10150(11)-4hrs. -02 10150(11)-4hrs. -02 Anneal Cycle Spray
Distance * Rate Furnace Temperature
in Rot % Pull in/min Chamber Holding 200 20 750 745 0.7-0.85 0.8-0.85 0.65-0.7 0.85 0.72 0.75 0.78 0.75 0.75 0.75 B Gear Set Hopper % 89-59 65 9 9 99 9 9 9 9 9 65 65 Current Gas Flow-CFH Amps Arc Powder 22-24 02 22 Ar 37 \$ 360 280 33-Dielectric ** LMTAF190-10A 24650-1 204-7A 23407-95 190-10A 191-10A 190-10A Solid 191-10A No preheat 665 LMTF475 G-8 Fines -177 + 74μ G-9 Fines -177 + 74 μ -177 + 74 H Number Ferrite 8-9 50% - 106 rpm 879 899 699 029 671 672 673 674 919 989 189 675 119 619 Date 8/11/77 8/15/77 8/9/77

Cracked just before dismount Blew off 1" from completion Substrate separation excessive to powder buildup at flame Increased "shooters" due New Ar and N₂ tanks -Graphite top plug No. 689, 690 Hot spot 1" from bottom Run not as good as 1-3 Broke in defurnacing Broke in defurnacing Broke in defurnacing Substrate separation Occasional hot spots Comment 10150(11)-4hrs. -02 10150(11)-4hrs. -02 Anneal Cycle Spray
Distance Rate Furnace Temperature
in Rot % Pull in/min Chamber Holding 200 200 740 730 740 740 750 750 765 765 0.75-0.8 0.75-0.80 0.75-0.78 0.8-0.75 0.8-0.75 0.7-0.78 0.7-0.65 0.65 0.75 0.92 0.78 0.75 8.0 8.0 B Gear Set Hopper Speed % 65-70 65-70 Current Gas Flow-CFH Amps Arc Powder 02 20 20 Ar 38 290-310 * 50% = 106 rpm ** No preheat *** Furnace turned off 290 315 295 290 280 330 325 LMTAF191-10A 315 Dielectric ** LMTF 200(1) 23407-95 LMTAF191-10A LMTAF207-7A 191-10A LMTF475 G-9 F 6-9 Fines -177 + 74 μ -177 + 74 pt Number Ferrite 685 687 688 689 88 683 584 069 692 663 694 969 169 8/15/77 Date 8/17/77

ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set

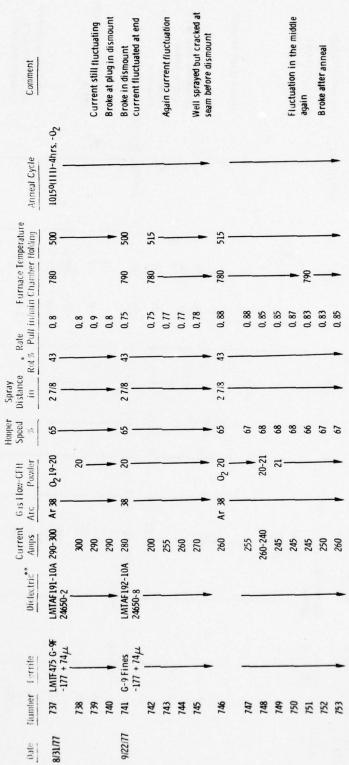


maintain deposit -Overlapped I" from bottom -Current fluctuations early in run Adjustments necessary to Broke at plug in dismount Broke at plugin dismount Jeposit erratic due to current fluctuations New cathode, Ar tank New holding furnace elements Current fluctuated Comment Powder low 10150(111)-4hrs. -02 10150(111)-4hrs. -02 Anneal Cycle Furnace Temperature istance * Rate Furnace Temperatur in Rot % Pull in/min Chamber Holding 200 790 770 790 780 022 780 780 800 0.75 0.88 0.75 0.75 0.78 0.65 0.75 0.9 0.8 0.72 0.73 0.73 0.7 0.8 ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set Spray Distance Hopper Speed 69-00 65-80 9-09 62 62 62 64 65 64 65 63 20-25 Powder Dielectric Amps Arc Powder 02 19 Ar 38 Ar 38 300-360 280-380 290-345 295-280 LMTAF191-10A 300 24650-2 290 290 290 290 290 280 300 280 LMTAF191-10A 285 24650-2 LMTF475 G-9F 6-9 Fines -177 + 74 μ -177 + 744 Number Ferrite 728 730 717 718 719 720 721 722 723 723 724 726 727 8/31/77 8/25/77

AIII-43

Date

ARC PLASMA LOG (Cont'd,) High Velocity Nozzle B Gear Set



Power fluctuated through-out run - broke in machin-ing Broke while cooling Comment 10150(111)-4hrs. -02 Anneal Cycle Spray
Distance Rate Furnace Temperature
in Rot's Pull in/min Chamber Holding 510 820-790 800 800 810 810 810 810 285 277 295 790 790 0.92 0.92 0.95 0.82 0.83 0.72 0.82 0.83 0.82 ARC PLASMA LOG (Cont'd.) High Velocity Nozzle B Gear Set 2 7/8 Hopper Speed % 9-09 60-63 19-59 63 67 65 63 65 57 20-21 02 20 Gas Flow-CFH Arc Powder Ar 38 Current 275 295 290 285 320 275 260 275 275 290 290 280 Uielectric** An 16-9 Fines LMIAF 192-10A 275 Number Ferrite 754 760 761 762 763 764

Date 9/23

*50% = 106 rpm ** No preheat

Seam crack, then broke in dismount

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APPENDIX IV

ELECTRONICS COMMAND TECHNICAL REQUIREMENTS SCS-478 30 September 1974

ARC PLASMA SPRAYED PHASE SHIFTER ELEMENTS

1. SCOPE

1.1 This specification establishes the manufacturing methods for the production of arc plasma sprayed ferrite phase shifter elements for C-band non-reciprocal waveguide phase shifters for phased array antennas.

2. APPLICABLE DOCUMENTS

MIL-STD-202 - Test Methods for Electronic and Electrical Component Parts.

3. REQUIREMENTS

- 3.1 Physical dimensions. The overall dimensions of the preliminary phase shifter element are illustrated in Figure 1.
- 3.1.1 Length. The length of the final production phase shifter element ferrite-dielectric composite shall be 5.145 inches.
- 3.1.2 Dielectric dimensions. The cross-sectional dimensions of the dielectric shall be 0.150 x 0.120 \pm 0.001 inches. The dielectric shall have a 0.040 x 0.020 inch hole the length of the dielectric, in the center of the insert. The hole which is required for the switching wires of the phase shifter may either be formed within the dielectric or by using two (2) dielectric halves, each with a cross-sectional dimension of 0.150 x 0.060 inches, in which a slot can be cut, to form the hole when the halves are placed together. The initial length of the dielectric shall have to be longer than 5.145 inches, in order that it extends beyond the ferrite deposit.
- 3.1.3 Ferrite dimensions. Ferrite shall be sprayed around the dielectric, such that the thickness of the deposit is enough to machine back to 0.050 \pm 0.001 inches on each side. To determine the proper spraying parameters of Paragraph 3.8, all spraying shall be conducted on the same dielectric as that used in the dielectric insert.

AD-A054 271

RAYTHEON CO WALTHAM MASS RESEARCH DIV
MANUFACTURING METHODS AND TECHNOLOGY MEASURE FOR ARC-PLASMA-SPR--ETC(U)
DEC 77 J J GREEN, H J VANHOOK, R J MAHER
DAAB07-75-C-0043
S-2287
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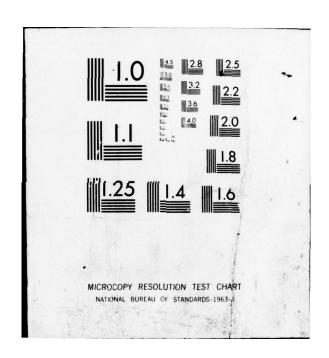






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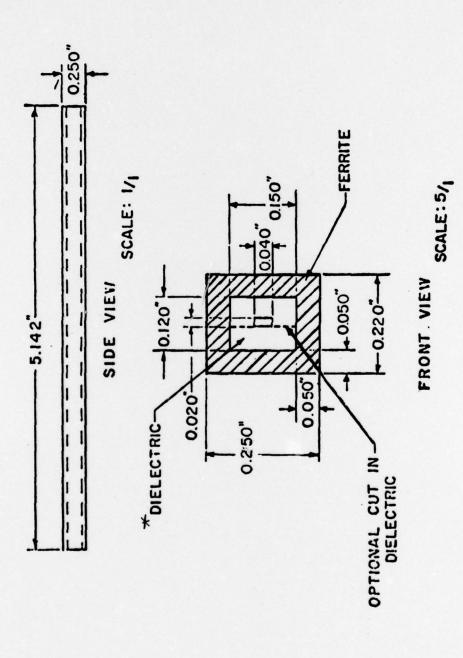
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- 3.2 Properties of dielectric. The dielectric shall have a loss tangent, $\tan \delta$, less than 0.0008, and a dielectric constant, K, greater than 18. The dielectric should exhibit a coefficient of linear expansion similar to that of the deposited ferrite.
- 3.3 Properties of the ferrite powder. The lithium ferrite powder to be sprayed, shall be a free flowing sprayed dried powder with the capability of high feed rates, greater than 10 gms/min. The conglomerate particle size should vary from 5 to 100 microns in size.
- 3.4 Properties of deposited ferrite. The magnetic properties of the arc plasma deposited ferrite shall exhibit the following characteristics:
- 3.4.1 The coercive force at room temperature shall be such that 90% of the required differential phase shift shall be obtained with at least 15 amperes of driving current.
- 3.4.2 Remanence at room temperature shall be such as to produce at least 340 degrees of differential phase shift, with driving current of 15 amperes.
- 3.4.3 Dielectric loss tangent of the ferrite at X-band shall be less than 0.0008.
- 3.4.4 Dielectric constant of the ferrite at X-band shall be greater than 15.
 - 3.4,5 Temperature dependence over the range of -30 to 85°C.
- 3.4.5.1 The remanence shall not vary more than $\pm 10\%$ over the temperature range.
- 3.4.5.2 The saturation magnetization shall not vary more than \pm 10% over the temperature range.
- 3.5 Physical characteristics of composite. The bond between the ferrite and the dielectric shall be such as to inhibit insertion loss spikes, and should not deteriorate over the temperature range of 3.4.5.
- 3.6 Physical handling. The phase shifter elements, after machining, shall be capable of withstanding normal physical handling during assembly to the test jig, during the measurements, and removal from test jig.

- 3.7 Device requirements. The following device requirements will be used as a guide to establish the reproducibility and yield of the device using the arc plasma spraying process, and are not intended to be the specifications of this program.
 - 3.7.1 Frequency 5.2 to 5.7 GHz.
 - 3.7.2 Insertion phase Mean + 160 at 5.45 GHz.
 - 3, 7, 3 Differential phase shift Mean + 100 at 5, 45 GHz.
- 3.7.4 Insertion loss less than 1.0 dB over the frequency range as specified in 3.7.1.
- 3.8 Arc plasma spraying parameters. The following arc plasma spraying parameters will be determined and recorded:
 - 3.8.1 Arc gas Type of arc gas or mixture and the flow rate.
 - 3.8.2 Carrier gas Type and flow rate.
 - 3.8.3 Working distance The distance from gun to dielectric.
 - 3.8.4 Powder feed Powder feed in lbs. /hr.
- 3.8.5 Oven temperature Temperature of oven at start and during spraying.
- 3.8.6 Other spraying variables Modification such as nozzle design, etc.
- 3.9 Device reproducibility. The measurements conducted under Paragraph 3.7 shall be used to establish the reproducibility of the arc plasma spraying process.
- 3.10 Microwave test fixture. A test fixture will be fabricated to accommodate the phase shifter element, such that each element can be located into this test fixture and the measurements of Paragraph 3.7 can be conducted. Appropriate transitions will be fabricated to match waveguide WR-187 (.872" x 1.872") to the test fixture (.250" x .750") to facilitate the testing required.
 - 4. QUALITY ASSURANCE PROVISIONS
 - 4.1 Inspection. -
- 4.1.1 Responsibility for inspection. The contractor is responsible for the performance of all inspections specified herein. The contractor may utilize his own facilities or any commercial laboratory acceptable to the Government. The tests shall be performed under the supervision of a technically qualified Government representative. Inspection records of the examinations and tests shall be kept complete and available to the Government.

- 4.2 Classification of inspection. Inspection shall be classified as follows:
- (a) First article inspection (does not include preparation for delivery) (See 4.3).
 - (b) Quality conformance inspection (See 4.4).
- 4.3 First article inspection. This inspection shall consist of all the tests included in the Government approved test procedure to show compliance with the requirements of Section 3. No failures shall be permitted.
 - 4.3.1 Schedule of tests. -
- (a) 20 each Determination of Remanence and Coercive Force at room temperature (See 3.4.1 and 3.4.2).
- (b) 10 each Determination of Remanence and Coercive Force over the temperature range (See 3, 4, 5).
- (c) 10 each (from b above) Determination of Insertion Loss, Insertion Phase, and Differential Phase Shift over the specified frequency range at room temperature.
- (d) 2 each (from c above) Determination of Insertion Loss, Insertion Phase, and Differential Phase Shift over the specified frequency and temperature ranges.
- 4.4 Quality conformance inspection. This inspection shall be performed on samples selected from the pilot production as specified in the bid request and contract.
 - 5. PREPARATION FOR DELIVERY
- 5.1 Preparation for delivery shall be in accordance with best commercial practices.



PBN-78-90

Figure 1 Arc Plasma C-Band Phase Shifter (Tolerance ± 0.001 in.).

* Dielectric core geometries of either center or exterior dots are acceptable.

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